New Directing Groups for Metal-Catalyzed Asymmetric Carbon–Carbon Bond-Forming Processes: Stereoconvergent Alkyl–Alkyl Suzuki Cross-Couplings of Unactivated Electrophiles

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Supporting Information

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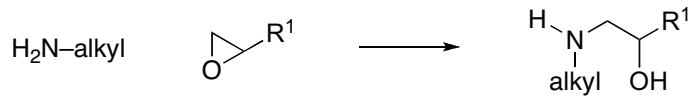
I. General

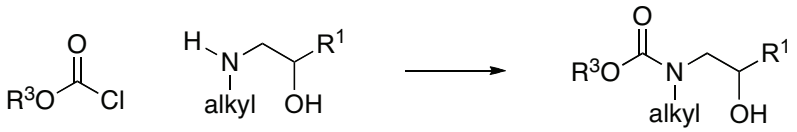
The following reagents were purchased and used as received: 9-BBN dimer (Aldrich), $NiBr_2$·diglyme (Aldrich; somewhat hygroscopic), diamine ligands (Aldrich), KO*t*-Bu (Acros or Strem), *n*-hexanol (Aldrich; anhydrous), *i*-BuOH (Aldrich; anhydrous), and *i*-Pr_2O (Aldrich; anhydrous). Unless otherwise noted, reactions were conducted with stirring in oven-dried glassware under an inert atmosphere. All NMR spectra were recorded on a 400 MHz spectrometer.

II. Preparation of Materials

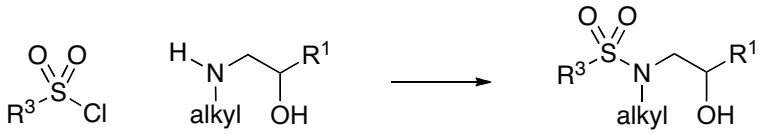
These procedures have not been optimized.



Representative Procedure A (synthesis of dialkylamines).[1] Calcium trifluoromethanesulfonate (30 mmol, 0.5 equiv) was added to a solution of the epoxide (60 mmol) and amine (60 mmol) in acetonitrile (150 mL) at 0 °C. The resulting mixture was stirred at room temperature overnight, and then the acetonitrile was removed by rotary evaporation. Water (50 ml) was added to the residue, and the mixture was extracted with CH_2Cl_2 (3 x 50 ml). The combined organic extracts were dried over $MgSO_4$ and then concentrated. The amino alcohol can be used without purification, or it can be purified by column chromatography.

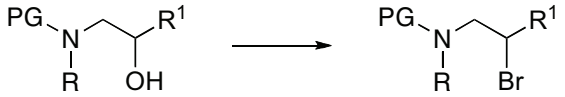


Representative Procedure B (synthesis of carbamate-containing electrophiles). Dry Et_2O (180 mL) and 2,6-lutidine (20 mmol) were added to a dry flask charged with the secondary amine (18 mmol). The reaction mixture was cooled to –78 °C, and then the chloroformate (18 mmol) was added. The mixture was allowed to warm to room temperature, and it was stirred overnight. Next, the reaction was quenched by the addition of aqueous HCl (1 M; 70 mL). The organic layer was separated, dried over $MgSO_4$, and then concentrated. The product was purified by column chromatography.

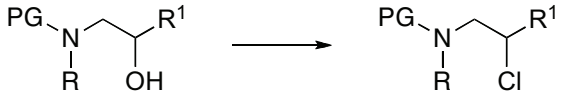


Representative Procedure C (synthesis of sulfonamide-containing electrophiles). Dry CH_2Cl_2 (18 mL) and 2,6-lutidine (3.95 mmol) were added to a dry flask was charged with the secondary amine (3.59 mmol). The reaction mixture was cooled to 0 °C, and then the sulfonyl chloride (3.59 mmol) was added. The mixture was allowed to warm to room temperature, and it was stirred overnight. Next, the reaction was quenched by the addition of aqueous HCl (1 M; 10 mL). The organic layer was separated, dried over $MgSO_4$, and then concentrated. The product was purified by column chromatography.

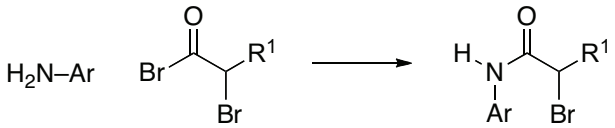
(1) Cepanec, I.; Litvić, M.; Mikuldaš, H.; Bartolincic, A.; Vinkovic, V. *Tetrahedron* **2003**, *59*, 2435–2439.



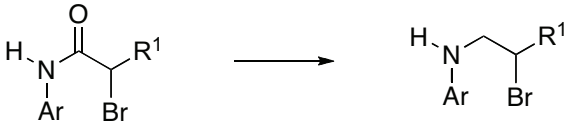
Representative Procedure D (synthesis of alkyl bromides). Dry CH_2Cl_2 (25 mL) and imidazole (2.1 mmol) were added to a dry flask charged with the alcohol (1.9 mmol). The reaction mixture was cooled to 0 °C, and then PPh_3Br_2 (2.1 mmol) was added. The mixture was allowed to warm to room temperature, and it was stirred overnight. Next, the reaction was quenched by the addition of water (15 mL). The organic layer was separated, dried over $MgSO_4$, and then concentrated. The product was purified by column chromatography.



Representative Procedure E (synthesis of alkyl chlorides). PPh_3 (4.0 mmol) and NCS (4.0 mmol) were added in turn to a stirred solution of the alcohol (3.6 mmol) in dry CH_2Cl_2 (20 mL) at 0 °C. The mixture was allowed to warm to room temperature, and it was stirred overnight. Next, the reaction was quenched by the addition of water (10 mL). The organic layer was separated, dried over $MgSO_4$, and then concentrated. The product was purified by column chromatography.



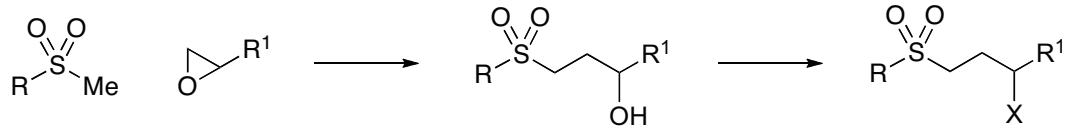
Representative Procedure F (synthesis of arylamines). Triethylamine (4.2 mL, 30 mmol) and the α-bromo acid bromide (20 mmol) were added dropwise in turn to a solution of the aniline (20 mmol) in CH_2Cl_2 (150 mL) at 0 °C. The mixture was allowed to warm to room temperature, and it was stirred for 2 h. Next, aqueous HCl (1 M; 50 mL) was added. The organic layer was separated, washed with water (50 mL) and brine (50 mL), dried over $MgSO_4$, filtered, and concentrated. The material may be used without purification in the next step.



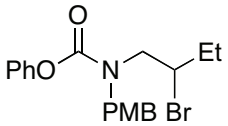
Representative procedure G (synthesis of arylamines). Borane–THF (1.0 M; 1.0 equiv) was added dropwise to a solution of the amide in dry THF (100 mL). After the addition was complete, the solution was heated at 50 °C for 18 h. Next, it was allowed to cool to room temperature, and then the reaction was quenched by the addition of water (10 mL). The THF

was removed by rotary evaporation, and the resulting mixture was diluted with Et_2O (100 mL). The organic layer was separated, washed with water (100 mL) and brine (100 mL), dried over $MgSO_4$, filtered, and concentrated. The material may be used without purification in the next step.

The arylamines were protected via reaction with chloroformates or sulfonyl chlorides, following Representative Procedures B and C.



Preparation of halo sulfones: The alcohols were prepared according to a literature procedure,[2] and they were converted to the bromide/chloride as described above.



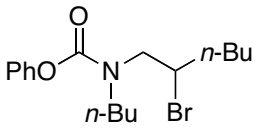
Phenyl 2-hydroxybutyl(4-methoxybenzyl)carbamate. The title compound was synthesized from phenyl 2-hydroxybutyl(4-methoxybenzyl)carbamate (1.00 g, 3.0 mmol) and triphenylphosphine dibromide (1.40 g, 3.3 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 0.87 g (74%) of a colorless oil.

^{1}H NMR ($CDCl_3$) δ 7.38–7.33 (m, 2H), 7.26–7.18 (m, 3H), 7.13–7.09 (m, 2H), 6.90–6.86 (m, 2H), 4.88–4.60 (m, 2H), 4.35–4.17 (m, 1H), 3.80 (s, 3H), 3.69–3.38 (m, 2H), 1.93–1.70 (m, 2H), 1.04 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 159.1, 155.0, 151.2, 129.6, 129.3, 128.7, 125.4, 121.6, 114.13, 114.08, 56.03, 55.56, 55.3, 54.2, 52.8, 51.8, 51.0, 29.5, 29.2, 12.0, 11.9 [mixture of rotamers].

FT-IR (film) 3018, 2936, 2838, 1717, 1613, 1513, 1457, 1409, 1203, 1114, 1035, 754, 668 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{19}H_{23}BrNO_3$: 392.0856, found: 392.0850.



Phenyl 2-bromohexyl(butyl)carbamate. The title compound was synthesized from phenyl butyl(2-hydroxyhexyl)carbamate (1.00 g, 3.4 mmol) and triphenylphosphine dibromide (1.58 g,

(2) Nwaukwa, S. O.; Lee, S.; Keehn, P. M. *Synth. Commun.* **1986**, *16*, 309–329.

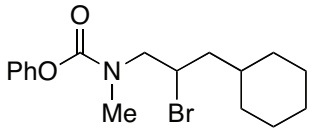
3.7 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 0.88 g (73%) of a colorless oil.

^1H NMR ($CDCl_3$) δ 7.36–7.33 (m, 2H), 7.20–7.18 (m, 1H), 7.10–7.08 (m, 2H), 4.39–4.23 (m, 1H), 3.83–3.58 (m, 2H), 3.45–3.37 (m, 2H), 1.94–1.73 (m, 2H), 1.67–1.55 (m, 3H), 1.46–1.26 (m, 5H), 0.97–0.87 (m, 6H).

^{13}C NMR ($CDCl_3$) δ 153.1, 151.3, 129.3, 125.3, 121.6, 55.4, 54.3, 54.0, 49.2, 48.7, 36.0, 35.8, 30.8, 29.6, 29.5, 22.1, 20.0, 13.9 [mixture of rotamers].

FT-IR (film) 3018, 2958, 2931, 1717, 1684, 1653, 1559, 1457, 1419, 1206, 755, 668 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{17}H_{27}BrNO_2$: 356.1220, found: 356.1217.



Phenyl 2-bromo-3-cyclohexylpropyl(methyl)carbamate. The title compound was synthesized from phenyl 3-cyclohexyl-2-hydroxypropyl(methyl)carbamate (1.50 g, 5.1 mmol) and triphenylphosphine dibromide (2.40 g, 5.7 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 1.29 g (72%) of a yellow solid.

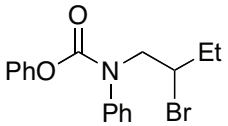
Mp: 60–62 °C.

^1H NMR ($CDCl_3$) δ 7.37–7.33 (m, 4H), 7.20–7.17 (m, 2H), 7.12–7.07 (m, 4H), 4.46–4.35 (m, 2H), 3.87–3.72 (m, 3H), 3.43–3.38 (m, 1H), 3.20 (s, 3H), 3.07 (s, 3H), 1.76–1.73 (m, 16H), 1.27–1.11 (m, 6H), 1.00–0.95 (m, 2H), 0.81–0.79 (m, 2H) [~1:1 mixture of rotamers].

^{13}C NMR ($CDCl_3$) δ 155.0, 154.5, 151.3, 151.2, 129.31, 129.26, 125.4, 125.3, 121.7, 121.6, 57.5, 56.6, 51.8, 51.1, 43.8, 43.6, 36.9, 36.2, 35.5, 35.4, 33.8, 33.6, 31.9, 31.7, 26.4, 26.1, 25.9 [mixture of rotamers].

FT-IR (film) 2922, 2851, 1723, 1594, 1449, 1394, 1205, 1131, 855, 750, 690 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{17}H_{25}BrNO_2$: 354.1063, found: 354.1072.



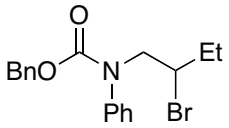
Phenyl 2-bromobutyl(phenyl)carbamate. The title compound was synthesized from *N*-(2-bromobutyl)aniline (0.70 g, 2.2 mmol) and phenyl chloroformate (0.30 mL, 2.2 mmol). Purification by chromatography (0%→50% Et_2O/hexanes) yielded 0.83 g (77%) of a clear oil.

^1H NMR ($CDCl_3$) δ 7.43–7.30 (m, 7H), 7.18–7.07 (m, 3H), 4.09–4.06 (m, 3H), 1.91–1.84 (m, 1H), 1.82–1.74 (m, 1H), 1.04 (t, 3H, J = 7.6 Hz).

^{13}C NMR ($CDCl_3$) δ 151.1, 129.2, 127.4, 125.4, 121.5, 65.8, 54.8, 31.5, 28.9, 22.6, 15.2, 14.1, 11.8.

FT-IR (film) 3064, 2969, 2936, 1725, 1596, 1495, 1456, 1390, 1325, 1294, 1206, 1164, 1137, 1072, 1025, 1004, 988, 925, 748, 699 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{17}H_{19}BrNO_2$: 348.0594, found: 348.0600.



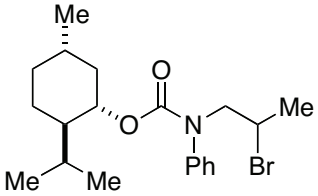
Benzyl 2-bromobutyl(phenyl)carbamate. The title compound was synthesized from *N*-(2-bromobutyl)aniline (1.40 g, 6.1 mmol) and benzyl chloroformate (0.90 mL, 6.1 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 1.40 g (64%) of a colorless oil.

^{1}H NMR ($CDCl_3$) δ 7.38–7.24 (m, 10H), 5.16 (d, 2H, J = 18.4 Hz), 4.13–3.95 (m, 3H), 1.93–1.67 (m, 2H), 1.00 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 155.4, 141.1, 136.3, 129.0, 128.3, 127.8, 127.4, 127.0, 67.3, 56.3, 55.0, 28.8, 11.7.

FT-IR (film) 3019, 2945, 2930, 2338, 1700, 1598, 1495, 1405, 1215, 757, 698, 668 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{18}H_{21}BrNO_2$: 362.0750, found: 362.0746.



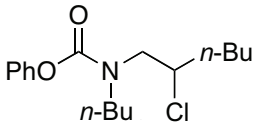
(1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl 2-bromopropyl(phenyl)carbamate. The title compound was synthesized from *N*-(2-bromobutyl)aniline (1.34 g, 6.1 mmol) and (*S*)-menthyl chloroformate (97% ee; 1.33 mL, 6.1 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 2.18 g (88%) of a light-yellow oil.

^{1}H NMR ($CDCl_3$) δ 7.36–7.32 (m, 2H), 7.25–7.20 (m, 3H), 4.58 (br s, 1H), 4.12–3.86 (m, 3H), 2.04 (br s, 1H), 1.79–1.44 (m, 7H), 1.02–0.73 (m, 13H) [mixture of diastereomers].

^{13}C NMR ($CDCl_3$) δ 155.4, 141.5, 128.9, 127.5, 126.8, 76.1, 57.6, 47.0, 46.5, 46.4, 41.1, 34.2, 31.3, 26.29, 26.27, 23.4, 23.2, 22.0, 20.7, 16.4 [mixture of diastereomers].

FT-IR (film) 2956, 2870, 1703, 1598, 1496, 1454, 1397, 1276, 1196, 1148, 1013, 966, 836, 766, 699 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{20}H_{31}BrNO_2$: 396.1533, found: 396.1553.



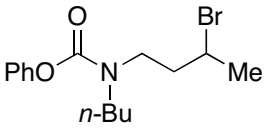
Phenyl butyl(2-chlorohexyl)carbamate. The title compound was synthesized from phenyl butyl(2-hydroxyhexyl)carbamate (1.00 g, 3.4 mmol) and NCS (0.50 g, 3.7 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 0.62 g (58%) of a colorless oil.

^{1}H NMR ($CDCl_3$) δ 7.37–7.33 (m, 2H), 7.20–7.16 (m, 1H), 7.10–7.08 (m, 2H), 4.29–4.15 (m, 1H), 3.78–3.23 (m, 4H), 1.87–1.26 (m, 10H), 0.97–0.87 (m, 6H).

^{13}C NMR ($CDCl_3$) δ 155.0, 154.3, 151.3, 151.2, 129.2, 125.3, 121.6, 61.1, 61.0, 55.0, 53.9, 49.3, 48.8, 35.5, 35.4, 30.8, 29.8, 28.4, 28.3, 22.2, 22.1, 20.0, 19.9, 13.9, 13.8 [mixture of rotamers].

FT-IR (film) 3018, 2958, 2936, 1717, 1653, 1595, 1496, 1465, 1413, 1206, 1163, 753, 690 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{17}H_{27}ClNO_2$: 312.1725, found: 312.1712.



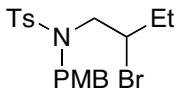
Phenyl 3-bromobutyl(butyl)carbamate. The title compound was synthesized from 4-(butylamino)butan-2-ol (2.00 g, 14 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 3.2 g (71% over 2 steps) of a light-yellow oil.

^{1}H NMR ($CDCl_3$) δ 7.36–7.32 (m, 2H), 7.17 (t, 1H, J = 7.4 Hz), 7.10–7.08 (m, 2H), 4.18–4.10 (m, 1H), 3.58–3.30 (m, 4H), 2.19–2.03 (m, 2H), 1.76–1.73 (m, 3H), 1.66–1.58 (m, 2H), 1.38–1.35 (m, 2H), 0.98–0.92 (m, 3H) [mixture of rotamers].

^{13}C NMR ($CDCl_3$) δ 154.7, 151.3, 129.2, 125.1, 121.6, 48.6, 48.0, 46.7, 46.0, 40.0, 39.2, 30.9, 30.1, 26.6, 26.5, 19.9, 13.8 [mixture of rotamers].

FT-IR (film) 3044, 2959, 2873, 1722, 1595, 1496, 1469, 1417, 1379, 1297, 1206, 1139, 1069, 1025, 1002, 911, 854, 753, 691, 616, 499 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{15}H_{23}BrNO_2$: 328.0907, found: 328.0928.



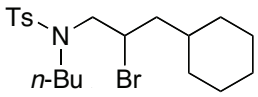
***N*-(2-Bromobutyl)-*N*-(4-methoxybenzyl)-4-methylbenzenesulfonamide.** The title compound was synthesized from *N*-(2-hydroxybutyl)-*N*-(4-methoxybenzyl)-4-methylbenzenesulfonamide (1.40 g, 3.8 mmol) and triphenylphosphine dibromide (1.79 g, 4.2 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 1.03 g (63%) of a white solid.

^{1}H NMR ($CDCl_3$) δ 7.71 (d, 2H, J = 8.4 Hz), 7.31 (d, 2H, J = 8.4 Hz), 7.17 (d, 2H, J = 8.4 Hz), 6.82 (d, 2H, J = 8.4 Hz), 4.40–4.15 (m, 2H), 3.77 (s, 3H), 3.41–3.34 (m, 1H), 3.08–2.93 (m, 2H), 2.42 (s, 3H), 1.29–1.22 (m, 2H), 0.76 (t, 3H, J = 7.6 Hz).

^{13}C NMR ($CDCl_3$) δ 159.5, 143.6, 136.1, 129.9, 129.8, 129.7, 129.5, 127.7, 127.3, 127.2, 114.0, 55.6, 55.2, 54.8, 53.3, 28.4, 21.5, 11.8 [mixture of rotamers].

FT-IR (film) 3020, 2967, 2805, 1612, 1513, 1456, 1339, 1252, 1160, 1090, 904, 814, 755, 667, 658 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{19}H_{25}BrNO_3S$: 428.0733, found: 428.0701.



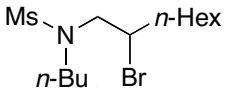
***N*-(2-Bromo-3-cyclohexylpropyl)-*N*-butyl-4-methylbenzenesulfonamide.** The title compound was synthesized from *N*-butyl-*N*-(3-cyclohexyl-2-hydroxypropyl)-4-methylbenzenesulfonamide (2.9 g, 7.9 mmol) and triphenylphosphine dibromide (3.3 g, 8.7 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 2.45 g (73%) of a light-yellow oil.

^{1}H NMR ($CDCl_3$) δ 7.67 (d, 4H, J = 8.4 Hz), 7.29 (d, 4H, J = 8.0 Hz), 4.26–4.19 (m, 2H), 3.70–3.69 (m, 1H), 3.54–3.42 (m, 2H), 3.32–3.27 (m, 2H), 3.21–3.13 (m, 2H), 3.07–3.00 (m, 2H), 2.40 (s, 3H), 2.39 (s, 3H), 1.82–0.86 (m, 39H) [~1:1 mixture of rotamers].

^{13}C NMR ($CDCl_3$) δ 143.4, 142.8, 141.2, 136.2, 129.7, 129.5, 127.3, 127.1, 121.9, 55.8, 51.7, 50.0, 35.5, 33.9, 32.6, 31.4, 30.6, 26.4, 26.2, 25.9, 25.8, 21.5, 19.9, 13.6 [mixture of rotamers].

FT-IR (film) 2925, 2852, 1599, 1494, 1449, 1342, 1305, 1222, 1160, 1120, 1091, 1030, 966, 922, 892, 866, 841, 815, 750, 728, 705, 657, 549 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{20}H_{33}BrNO_2S$: 432.1402, found: 432.1409.



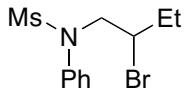
***N*-(2-Bromooctyl)-*N*-butylmethanesulfonamide.** The title compound was synthesized from *N*-butyl-*N*-(2-hydroxyoctyl)methanesulfonamide (3.70 g, 13.2 mmol) and triphenylphosphine dibromide (6.10 g, 14.5 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 2.10 g (46%) of a colorless oil.

^{1}H NMR ($CDCl_3$) δ 4.17–4.10 (m, 1H), 3.57–3.38 (m, 2H), 3.32–3.10 (m, 2H) 2.87 (s, 3H), 1.95–1.87 (m, 1H), 1.72–1.24 (m, 13H), 0.93–0.83 (m, 6H).

^{13}C NMR ($CDCl_3$) δ 54.6, 54.2, 49.0, 39.0, 35.7, 31.5, 30.5, 28.5, 27.3, 22.5, 19.9, 14.0, 13.7.

FT-IR (film) 3020, 2953, 2932, 1653, 1559, 1457, 1335, 1216, 1148, 759, 668 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{13}H_{29}BrNO_2S$: 344.1097, found: 344.1068.



***N*-(2-Bromobutyl)-*N*-phenylmethanesulfonamide.** The title compound was synthesized from *N*-(2-bromobutyl)aniline (0.70 g, 3.1 mmol) and methanesulfonyl chloride (0.40 mL, 4.6 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 0.193 g (20%) of a white solid.

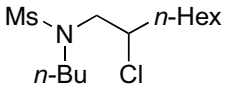
Mp: 82–84 °C.

^{1}H NMR ($CDCl_3$) δ 7.43–7.33 (m, 5H), 4.07–4.02 (m, 1H), 3.95–3.80 (m, 2H), 2.91 (s, 3H), 2.04–1.96 (m, 1H), 1.76–1.67 (m, 1H), 0.99 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 139.0, 129.7, 128.6, 128.5, 56.9, 54.9, 37.6, 28.3, 11.4.

FT-IR (film) 2971, 2935, 1595, 1493, 1455, 1342, 1222, 1192, 1155, 1070, 1027, 1005, 961, 879, 776, 697, 544, 522 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{11}H_{16}BrNNaO_2S$: 329.9977, found: 329.9971.



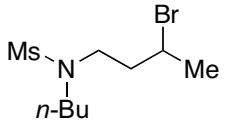
***N*-(2-Chloroctyl)-*N*-butylmethanesulfonamide.** The title compound was synthesized from *N*-butyl-*N*-(2-hydroxyoctyl)methanesulfonamide (1.00 g, 3.6 mmol) and NCS (0.53 g, 4.0 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 0.60 g (56%) of a colorless oil.

^{1}H NMR ($CDCl_3$) δ 4.09–4.03 (m, 1H), 3.49–3.28 (m, 4H), 2.87 (s, 3H), 1.75–1.63 (m, 1H), 1.51–1.25 (m, 13H), 0.90 (t, 3H, J = 7.2 Hz), 0.85 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 61.0, 54.1, 48.9, 39.1, 35.5, 31.6, 30.5, 28.7, 26.1, 22.5, 19.9, 14.0, 13.7.

FT-IR (film) 3020, 2955, 2932, 1333, 1215, 1149, 758, 668 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{13}H_{29}ClNO_2S$: 298.1602, found: 298.1606.



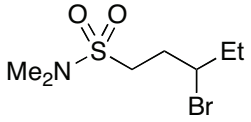
***N*-(3-Bromobutyl)-*N*-butylmethanesulfonamide.** The title compound was synthesized from 4-(*N*-butylmethylsulfonamido)butan-2-yl methanesulfonate (2.42 g, 14 mmol) and LiBr (2.8 g, 32 mmol) in acetone. Purification by chromatography (0%→50% Et_2O/hexanes) yielded 1.72 g (61%) of a light-yellow oil.

^{1}H NMR ($CDCl_3$) δ 4.14–4.06 (m, 1H), 3.35–3.33 (m, 2H), 3.24–3.13 (m, 2H), 2.81 (s, 3H), 2.16–1.97 (m, 2H), 1.71 (d, 3H, J = 6.4 Hz), 1.62–1.53 (m, 2H), 1.36–1.27 (m, 2H), 0.91 (t, 3H, J = 7.4 Hz).

^{13}C NMR ($CDCl_3$) δ 48.5, 48.1, 46.7, 40.5, 37.8, 30.7, 26.5, 19.8, 13.7.

FT-IR (film) 2960, 2933, 2873, 1458, 1379, 1332, 1241, 1203, 1147, 1047, 961, 928, 880, 774, 705, 520 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_9H_{20}BrNNaO_2S$: 310.0276, found: 310.0278.



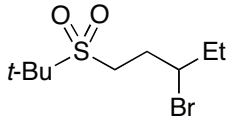
3-Bromo-*N*,*N*-dimethylpentane-1-sulfonamide. The title compound was synthesized from 3-hydroxy-*N*,*N*-dimethylpentane-1-sulfonamide[3] (2.00 g, 10.2 mmol) and triphenylphosphine dibromide (4.80 g, 11.4 mmol). Purification by chromatography (30%→50% Et_2O/hexanes) yielded 2.16 g (82%) of a colorless oil.

^1H NMR ($CDCl_3$) δ 4.07–4.00 (m, 1H), 3.23–3.16 (m, 1H), 3.06–2.99 (m, 1H), 2.87 (s, 6H), 2.39–2.30 (m, 1H), 2.30–2.14 (m, 1H), 1.91–1.83 (m, 2H), 1.04 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 57.3, 46.5, 37.5, 32.3, 32.1, 12.0.

FT-IR (film) 2969, 1459, 1333, 1143, 959, 748, 709 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_7H_{16}BrNNaO_2S$: 281.9961, found: 281.9967.



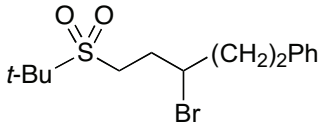
3-Bromo-1-(*tert*-butylsulfonyl)pentane. The title compound was synthesized from 1-(*tert*-butylsulfonyl)pentan-3-ol (1.50 g, 7.2 mmol) and triphenylphosphine dibromide (3.3 g, 7.9 mmol). Purification by chromatography (10%→100% EtOAc/hexanes) yielded 1.48 g (76%) of a colorless liquid.

^1H NMR ($CDCl_3$) δ 4.03–3.97 (m, 1H), 3.16–3.09 (m, 1H), 2.99–2.92 (m, 1H), 2.37–2.29 (m, 1H), 2.20–2.10 (m, 1H), 1.84–1.73 (m, 2H), 1.31 (s, 9H), 0.96 (t, 3H, J = 7.4 Hz).

^{13}C NMR ($CDCl_3$) δ 58.8, 57.6, 43.9, 32.1, 29.5, 23.1, 11.7.

FT-IR (film) 2972, 2878, 1464, 1398, 1366, 1294, 1211, 1116, 1050, 1019, 953, 903, 806, 755, 722, 664, 601, 536 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_9H_{19}BrNaO_2S$: 293.0181, found: 293.0198.



(3-Bromo-5-(*tert*-butylsulfonyl)pentyl)benzene. The title compound was synthesized from 1-(*tert*-butylsulfonyl)-5-phenylpentan-3-ol (1.5 g, 5.3 mmol) and triphenylphosphine dibromide

(3) Synthesized according to: Nwaukwa, S. O.; Lee, S.; Keehn, P. M. *Synth. Commun.* **1986**, *16*, 309–329.

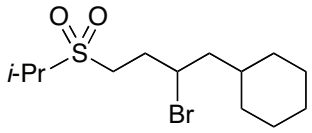
(2.5 g, 5.8 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 1.53 g (83%) of a white solid.

^1H NMR ($CDCl_3$) δ 7.30–7.27 (m, 2H), 7.21–7.18 (m, 3H), 4.07–4.01 (m, 1H), 3.27–3.20 (m, 1H), 3.03–2.96 (m, 1H), 2.94–2.87 (m, 1H), 2.80–2.73 (m, 1H), 2.50–2.41 (m, 1H), 2.35–2.12 (m, 3H), 1.41 (s, 9H).

^{13}C NMR ($CDCl_3$) δ 140.3, 128.6, 128.5, 126.3, 59.2, 55.1, 44.2, 40.8, 33.5, 30.1, 23.4.

FT-IR (film) 3062, 3027, 2939, 1603, 1496, 1455, 1398, 1366, 1287, 1205, 1115, 1017, 986, 806, 750, 722, 701, 665 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{15}H_{23}BrNaO_2S$: 369.0494, found: 369.0516.



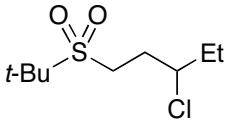
(2-Bromo-4-(isopropylsulfonyl)butyl)cyclohexane. The title compound was synthesized from 1-cyclohexyl-4-(isopropylsulfonyl)butan-2-ol (2.0 g, 7.6 mmol) and triphenylphosphine dibromide (3.5 g, 8.3 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 1.44 g (56%) of a white solid.

^1H NMR ($CDCl_3$) δ 4.20–4.14 (m, 1H), 3.28–3.21 (m, 1H), 3.15–3.00 (m, 2H), 2.45–2.36 (m, 1H), 2.23–2.13 (m, 1H), 1.85–1.78 (m, 1H), 1.74–1.54 (m, 7H), 1.39 (d, 6H, J = 6.8 Hz), 1.29–1.07 (m, 3H), 0.98–0.90 (m, 1H), 0.84–0.76 (m, 1H).

^{13}C NMR ($CDCl_3$) δ 53.31, 53.28, 47.5, 46.8, 35.5, 33.4, 32.0, 30.9, 26.3, 26.0, 25.9, 15.5, 15.1.

FT-IR (film) 2924, 2852, 1448, 1308, 1231, 1169, 1122, 1052, 964, 879, 839, 758, 689 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{13}H_{25}BrNaO_2S$: 347.0651, found: 347.0662.



1-(*tert*-Butylsulfonyl)-3-chloropentane. The title compound was synthesized from 1-(*tert*-butylsulfonyl)pentan-3-ol (1.50 g, 7.2 mmol), NCS (1.10 g, 8.2 mmol), and triphenylphosphine (2.10 g, 8.0 mmol). Purification by chromatography (5%→100% Et_2O/hexanes) yielded 1.24 g (75%) of a white solid.

Mp: 32–34 °C.

^1H NMR ($CDCl_3$) δ 3.93–3.86 (m, 1H), 3.14–3.07 (m, 1H), 2.98–2.90 (m, 1H), 2.31–2.23 (m, 1H), 2.09–1.99 (m, 1H), 1.80–1.61 (m, 2H), 1.31 (s, 9H), 0.94 (t, 3H, J = 7.4 Hz).

^{13}C NMR ($CDCl_3$) δ 63.5, 58.8, 42.8, 31.4, 28.9, 23.1, 10.6.

FT-IR (film) 2973, 2940, 2879, 1464, 1399, 1366, 1293, 1200, 1116, 1051, 1019, 958, 906, 860, 808, 756, 723, 666, 601, 544 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_9H_{19}ClNaO_2S$: 249.0686, found: 249.0698.

III. Stereoconvergent Suzuki Cross-Coupling Reactions

General Procedure (carbamates) (conducted in a glovebox; however, see the note below). The trialkylborane was prepared by adding 9-BBN dimer (2.28 g, 9.3 mmol), *i*-Pr_2O (5.0 mL), and the alkene (18.7 mmol) in turn to a 20-mL vial. The vial was capped and removed from the glovebox. The reaction mixture was stirred at 60 °C for 1.5 h, and then it was allowed to cool to r.t. The vial was taken back into the glovebox, and the reaction mixture was diluted with *i*-Pr_2O to furnish a 1.5 M solution. Next, a portion of this solution (0.67 mL, 1.00 mmol) was added to a solution of KO*t*-Bu (78 mg, 0.70 mmol) in *n*-hexanol (113 μL, 0.90 mmol) in a 4-mL vial. The resulting mixture was stirred at r.t. for 45 min.

A solution of $NiBr_2$·diglyme (17.6 mg, 0.050 mmol) and (1*R*,2*R*)-(+)-*N*,*N*'-dimethyl-1,2-diphenyl-1,2-ethanediamine ((*R*,*R*)-DMPEDA; 14.7 mg, 0.061 mmol) in *i*-Pr_2O (2.5 mL) in a 20-mL vial was stirred at r.t. for 30 min. Next, a solution of the electrophile (0.50 mmol) in *i*-Pr_2O (2.0 mL) was added, along with an *i*-Pr_2O rinsing (0.5 mL) (Note: if the electrophile is not soluble in *i*-Pr_2O, then it was added as a solid, followed by 2.5 mL of *i*-Pr_2O), and then the solution of the activated trialkylborane was added dropwise over 30 seconds. The reaction mixture was stirred at r.t. for 72 h, and then it was filtered through silica gel, eluting with Et_2O (20 mL). The solvent was removed by rotary evaporation, and the residue was diluted with hexanes (10 mL). The resulting solution was filtered through an acrodisc and then concentrated by rotary evaporation.

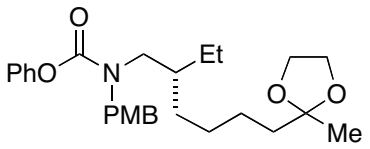
A second run was performed with the (1*S*,2*S*) enantiomer ((*S*,*S*)-DMPEDA) of the ligand.

General Procedure (sulfonamides and sulfones) (conducted in a glovebox; however, see the note below). The trialkylborane was prepared by adding 9-BBN dimer (2.28 g, 9.3 mmol), *i*-Pr_2O (5.0 mL), and the alkene (18.7 mmol) in turn to a 20-mL vial. The vial was capped and removed from the glovebox. The reaction mixture was stirred at 60 °C for 1.5 h, and then it was allowed to cool to r.t. The vial was taken back into the glovebox, and the reaction mixture was diluted with *i*-Pr_2O to furnish a 1.5 M solution. Next, a portion of this solution (0.67 mL, 1.00 mmol) was added to a solution of KO*t*-Bu (78 mg, 0.70 mmol) in *i*-butanol (83 μL, 0.90 mmol) in a 4-mL vial. The resulting mixture was stirred at r.t. for 45 min.

A solution of $NiBr_2$·diglyme (17.6 mg, 0.050 mmol) and (1*R*,2*R*)-(+)-*N*,*N*'-dimethyl-1,2-bis[(3-trifluoromethyl)phenyl]-1,2-ethanediamine ((*R*,*R*)-*m*-CF_3-DMPEDA; 22.6 mg, 0.060 mmol) in *i*-Pr_2O (2.5 mL) in a 20-mL vial was stirred at r.t. for 30 min. Next, a solution of the electrophile (0.50 mmol) in *i*-Pr_2O (2.0 mL) was added, along with an *i*-Pr_2O rinsing (0.5 mL) (Note: if the electrophile is not soluble in *i*-Pr_2O, then it was added as a solid, followed by 2.5 mL of *i*-Pr_2O), and then the solution of the activated trialkylborane was added dropwise over 30 seconds. The reaction mixture was stirred at r.t. for 72 h, and then it was filtered through silica gel, eluting with Et_2O (20 mL). The solvent was removed by rotary evaporation, and the residue was diluted with hexanes (10 mL). The resulting solution was filtered through an acrodisc and then concentrated by rotary evaporation.

A second run was performed with the (1*S*,2*S*) enantiomer ((*S*,*S*)-*m*-CF_3-DMPEDA) of the ligand.

Note: For the sake of convenience, the stereoconvergent Suzuki cross-couplings were conducted in a glovebox. However, this method does *not* require the use of a glovebox. When carried out using Schlenk techniques without a glovebox, the coupling illustrated in entry 3 of Table 2 proceeded in 92% ee and 89% yield.



(*R*)-Phenyl 2-ethyl-6-(2-methyl-1,3-dioxolan-2-yl)hexyl(4-methoxybenzyl)carbamate (Table 1, entry 1). Phenyl 2-hydroxybutyl(4-methoxybenzyl)carbamate (196 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 2-(but-3-enyl)-2-methyl-1,3-dioxolane with 9-BBN dimer (1.5 M in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 125 mg (55%, 92% ee). Second run: 128 mg (56%, 90% ee).

The ee was determined by HPLC on an AD-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 28.8 min (minor), 30.1 min (major).

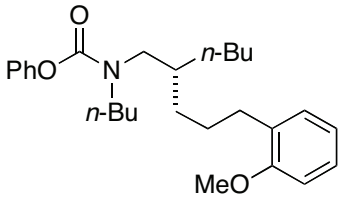
^{1}H NMR ($CDCl_3$) δ 7.36–7.33 (m, 2H), 7.25–7.07 (m, 5H), 6.88–6.85 (m, 2H), 4.55 (s, 1H), 4.48 (s, 1H), 3.93–3.89 (m, 4H), 3.80 (s, 3H), 3.14–3.13 (m, 2H), 1.72–1.65 (m, 1H), 1.61–1.20 (m, 13H), 0.86 (t, 3H, *J* = 7.6 Hz) [mixture of rotamers].

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 155.5, 155.0, 151.5, 129.5, 129.2, 128.6, 125.1, 121.7, 121.6, 113.9, 110.0, 64.6, 55.2, 50.4, 50.2, 50.0, 49.5, 39.1, 37.7, 37.4, 30.8, 29.7, 26.6, 24.4, 23.8, 23.7, 10.6 [mixture of rotamers].

FT-IR (film) 3016, 2936, 1717, 1708, 1615, 1513, 1457, 1418, 1205, 1037, 734, 667, 649 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{27}H_{38}NO_5$: 456.2744, found: 456.2755.

$[\alpha]^{22}_D$ = –2.4 (c 1.0, $CHCl_3$) obtained with (*R,R*)-DMPEDA.



(*R*)-Phenyl butyl(2-(3-(2-methoxyphenyl)propyl)hexyl)carbamate (Table 1, entry 2). Phenyl 2-bromohexyl(butyl)carbamate (178 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 177 mg (83%, 90% ee). Second run: 176 mg (83%, 89% ee).

The ee was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 9.6 min (major), 16.0 min (minor).

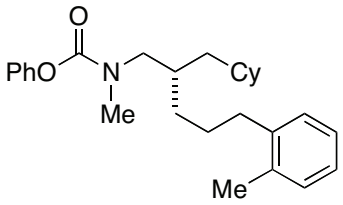
^{1}H NMR ($CDCl_3$) δ 7.36–7.32 (m, 2H), 7.19–7.12 (m, 5H), 6.89–6.82 (m, 2H), 3.80 (s, 3H), 3.38–3.18 (m, 4H), 2.60 (t, 2H, *J* = 7.6 Hz), 1.79–1.66 (m, 1H), 1.65–1.55 (m, 4H), 1.40–1.25 (m, 10H), 0.97–0.87 (m, 6H).

^{13}C NMR ($CDCl_3$) δ 157.3, 154.8, 151.5, 130.9, 130.8, 129.7, 129.1, 126.9, 126.8, 124.9, 121.70, 121.66, 120.3, 110.2, 55.1, 51.6, 51.1, 47.7, 47.5, 36.7, 36.2, 31.3, 31.2, 31.0, 30.6, 30.5, 29.7, 28.6, 26.7, 23.1, 20.1, 20.0, 14.1, 13.8 [mixture of rotamers].

FT-IR (film) 3019, 2950, 2936, 1717, 1700, 1653, 1559, 1457, 1419, 1215, 758, 669 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{27}H_{40}NO_3$: 426.3003, found: 426.3016.

$[\alpha]^{22}_D$ = –2.1 (c 1.0, $CHCl_3$) obtained with (*S*,*S*)-DMPEDA.



(*S*)-Phenyl 2-(cyclohexylmethyl)-5-*o*-tolylpentyl(methyl)carbamate (Table 1, entry 3). Phenyl 2-bromo-3-cyclohexylpropyl(methyl)carbamate (180 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methylbenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 149 mg (72%, 89% ee). Second run: 158 mg (76%, 90% ee).

The ee was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 13.9 min (major), 16.4 min (minor).

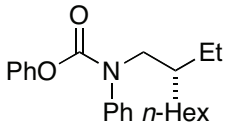
^{1}H NMR ($CDCl_3$) δ 7.40–7.36 (m, 4H), 7.23–7.10 (m, 14H), 3.40–3.26 (m, 4H), 3.07 (s, 3H), 3.00 (s, 3H), 2.62 (t, 4H, *J* = 7.8 Hz), 2.34 (s, 3H), 2.32 (s, 3H), 1.95–1.14 (m, 32H), 1.00–0.84 (m, 4H) [~1:1 mixture of rotamers].

^{13}C NMR ($CDCl_3$) δ 155.0, 151.5, 140.9, 140.6, 140.5, 135.7, 135.6, 130.1, 130.03, 129.98 , 129.9, 129.14, 129.10, 128.7, 125.8, 125.7, 125.6, 125.5, 125.02, 124.97, 121.7, 121.6, 53.9, 53.6, 39.7, 39.6, 35.3, 35.1, 34.9, 34.8, 33.9, 33.8, 33.6, 33.4, 33.3, 33.2, 32.9, 31.9, 31.7, 30.2, 29.5, 26.8, 26.5 [mixture of rotamers].

FT-IR (film) 3016, 2922, 2851, 1725, 1595, 1494, 1449, 1400, 1293, 1206, 1164, 1128, 1026, 908, 854, 748, 691 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{27}H_{38}NO_2$: 408.2897, found: 408.2896.

$[\alpha]^{24}_D$ = +4.8° (c 0.50, CH_2Cl_2) obtained with (*R*,*R*)-DMPEDA.



(*R*)-Phenyl 2-ethyloctyl(phenyl)carbamate (Table 1, entry 4). Phenyl 2-bromobutyl(phenyl)carbamate (175 mg, 0.50 mmol) and a solution of the reagent prepared by hydroboration of 1-hexene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 102 mg (57%, 90% ee). Second run: 97 mg (54%, 91% ee).

The ee was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 9.2 min (minor), 10.2 min (major).

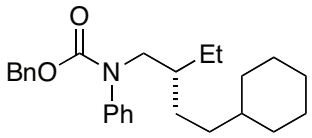
^{1}H NMR ($CDCl_3$) δ 7.39–7.29 (m, 7H), 7.16–7.06 (m, 3H), 3.74–3.71 (m, 2H), 1.52–1.19 (m, 13H), 0.86–0.80 (m, 6H).

^{13}C NMR ($CDCl_3$) δ 154.3, 151.4, 141.7, 129.2, 129.1, 129.0, 127.3, 126.8, 125.2, 121.6, 53.9, 31.8, 30.5, 29.6, 26.2, 23.5, 22.6, 14.1, 10.4.

FT-IR (film) 3043, 2928, 2857, 1718, 1597, 1495, 1457, 1394, 1274, 1204, 1164, 1133, 1072, 1026, 1006, 749, 699, 638 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{23}H_{32}NO_2$: 354.2428, found: 354.2423.

$[\alpha]^{25}_D$ = –7.6° (c 0.25, CH_2Cl_2) obtained with (*R*,*R*)-DMPEDA.



(*R*)-Benzyl 4-cyclohexyl-2-ethylbutyl(phenyl)carbamate (Table 1, entry 5). Benzyl 2-bromobutyl(phenyl)carbamate (181 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of vinylcyclohexane with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 130 mg (66%, 79% ee). Second run: 132 mg (66%, 82% ee).

The ee was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 9.6 min (minor), 10.6 min (major).

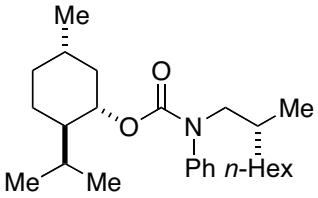
^{1}H NMR ($CDCl_3$) δ 7.36–7.18 (m, 10H), 5.14 (s, 2H), 3.62 (d, 2H, *J* = 7.2 Hz), 1.63–1.04 (m, 18H), 0.77 (t, 3H, *J* = 7.6 Hz).

^{13}C NMR ($CDCl_3$) δ 155.7, 136.7, 128.8, 128.3, 127.7, 127.3, 126.4, 67.0, 53.3, 37.9, 33.8, 33.31, 33.26, 27.5, 26.6, 26.3, 23.4, 10.3 [mixture of rotamers].

FT-IR (film) 3014, 2923, 2851, 1701, 1597, 1496, 1456, 1404, 1274, 1216, 1148, 1015, 756, 697, 667 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{26}H_{36}NO_2$: 394.2741, found: 394.2736.

$[\alpha]^{22}_D$ = +6.5° (c 1.0, $CHCl_3$) obtained with (*R*,*R*)-DMPEDA.



(1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl (*R*)-2-methyloctyl(phenyl)carbamate (eq 2). (1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl 2-bromopropyl(phenyl)carbamate (200 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of hexene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. Run with (*R*,*R*) ligand: 116 mg (57%, 79% de).

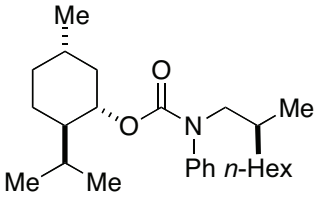
The de was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 6.5 min (minor), 7.1 min (major).

^{1}H NMR ($CDCl_3$) δ 7.33–7.29 (m, 2H), 7.20–7.17 (m, 3H), 4.58–4.54 (m, 1H), 3.61–3.44 (m, 2H), 2.07–2.04 (m, 1H), 1.82–1.80 (m, 1H), 1.60–0.96 (m, 15H), 0.85–0.74 (m, 18H) [mixture of rotamers].

^{13}C NMR ($CDCl_3$) δ 155.7, 142.3, 128.7, 127.3, 126.1, 75.5, 55.9, 47.1, 41.2, 34.3, 34.1, 32.0, 31.8, 31.3, 29.5, 26.7, 26.2, 23.4, 22.6, 22.0, 20.7, 17.3, 16.4, 14.1.

FT-IR (film) 2956, 2927, 2871, 1702, 1598, 1497, 1457, 1400, 1293, 1274, 1148, 1013, 766, 698 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{26}H_{43}NNaO_2$: 424.3186, found: 424.3191.



(1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl (*S*)-2-methyloctyl(phenyl)carbamate (eq 3). (1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl 2-bromopropyl(phenyl)carbamate (200 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-hexene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. Run with (*S*,*S*) ligand: 110 mg (54%, 80% de).

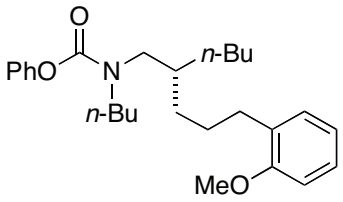
The de was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 6.3 min (major), 6.9 min (minor).

^{1}H NMR ($CDCl_3$) δ 7.33–7.29 (m, 2H), 7.20–7.15 (m, 3H), 4.58–4.54 (m, 1H), 3.61–3.45 (m, 2H), 2.07–2.05 (m, 1H), 1.82–1.80 (m, 1H), 1.60–1.00 (m, 15H), 0.87–0.74 (m, 18H) [mixture of rotamers].

^{13}C NMR ($CDCl_3$) δ 155.7, 142.3, 128.7, 127.3, 126.1, 55.8, 47.1, 41.2, 34.3, 34.0, 32.0, 31.8, 31.3, 29.5, 26.7, 26.3, 23.4, 22.6, 22.0, 20.7, 17.3, 16.4.

FT-IR (film) 3040, 2925, 2871, 1705, 1598, 1497, 1456, 1400, 1343, 1274, 1181, 1148, 1080, 1039, 1013, 997, 984, 968, 920, 879, 847, 766, 698, 649 cm^{-1}.

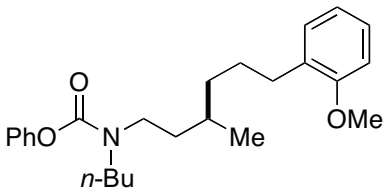
MS (ESI) $[M+H]^+$ calcd for $C_{26}H_{44}NO_2$: 402.3367, found: 402.3375.



(*R*)-Phenyl butyl(2-(3-(2-methoxyphenyl)propyl)hexyl)carbamate (eq 4). Phenyl 2-chlorohexyl(butyl)carbamate (156 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 166 mg (78%, 90% ee). Second run: 172 mg (81%, 89% ee).

The ee was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 9.6 min (major), 16.0 min (minor).

For characterization data, see Table 1, entry 2 (above).



(*R*)-Phenyl butyl(6-(2-methoxyphenyl)-3-methylhexyl)carbamate (eq 5). Phenyl 3-bromobutyl(butyl)carbamate (160 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 184 mg (94%, 52% ee). Second run: 184 mg (94%, 55% ee). Third run with (1*R*,2*R*)-(+)-*N*,*N*′-dimethyl-1,2-bis[(3-trifluoromethyl)phenyl]-1,2-ethanediamine: 174 mg (89%, 76% ee).

The ee was determined by HPLC on an OD-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 9.8 min (minor), 11.1 min (major).

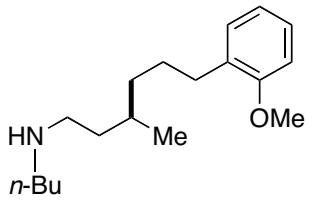
^{1}H NMR ($CDCl_3$) δ 7.40–7.36 (m, 2H), 7.23–7.15 (m, 5H), 6.91–6.86 (m, 2H), 3.82 (s, 3H), 3.42–3.35 (m, 4H), 2.66–2.63 (m, 2H), 1.86–1.30 (m, 11H), 0.99 (d, 6H, *J* = 5.6 Hz) [mixture of rotamers].

^{13}C NMR ($CDCl_3$) δ 157.3, 154.4, 151.5, 131.0, 130.8, 129.6, 129.0, 126.7, 124.8, 121.6, 120.2, 110.0, 55.0, 47.3, 47.0, 45.9, 45.5, 36.7, 36.6, 35.6, 34.8, 30.8, 30.5, 30.4, 30.2, 30.0, 27.1, 19.9, 19.5, 19.4, 13.8 [mixture of rotamers].

FT-IR (film) 3426, 2932, 2049, 1716, 1599, 1494, 1378, 1205, 1031, 942, 909, 887, 853, 751, 690 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{25}H_{36}NO_3$: 398.2690, found: 398.2695.

$[\alpha]^{24}_D = -11.5°$ (c 0.50, CH_2Cl_2) obtained with (*R*,*R*)-*m*-CF_3-DMPEDA.



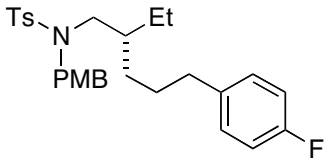
(*R*)-*N*-butyl-6-(2-methoxyphenyl)-3-methylhexan-1-amine (eq 6). A solution of (*R*)-phenyl butyl(6-(2-methoxyphenyl)-3-methylhexyl)carbamate (176 mg, 0.46 mmol) in DMSO (2.3 mL) and aqueous KOH (6 M; 2.3 mL) in a 20 mL-vial was heated at 95 °C for 24 h. Next, the reaction mixture was cooled to r.t., diluted with water (20 mL), and extracted with Et_2O (3 x 20 mL). The combined organic layers were washed with brine (4 x 20 mL), and the solvent was removed. CH_2Br_2 was added as an internal standard, and the yield (83%) was determined by ^1H NMR spectroscopy.

The amine can be purified and isolated as the *p*-toluenesulfonic acid salt: The unpurified amine was dissolved in Et_2O (11.5 mL), and *p*-toluenesulfonic acid monohydrate (96 mg, 0.50 mmol) was added. The mixture was stirred at r.t. for 1.75 h, and then it was concentrated, and the residue was purified by flash column chromatography (5% MeOH/CH_2Cl_2→20% MeOH/CH_2Cl_2). Beige powder. First run: 147 mg (71%). Second run: 156 mg (76%).

^1H NMR ($CDCl_3$) δ 8.65 (br s, 2H), 7.71 (d, 2H, *J* = 8.0 Hz), 7.16–7.06 (m, 4H), 6.86–6.79 (m, 2H), 3.77 (s, 3H), 2.93–2.81 (m, 4H), 2.58–2.44 (m, 2H), 2.31 (s, 3H), 1.75–1.68 (m, 3H), 1.59–1.41 (m, 4H), 1.33–1.24 (m, 3H), 1.14–1.04 (m, 1H), 0.86 (t, 3H, *J* = 7.4 Hz), 0.80 (d, 3H, *J* = 6.4 Hz).

^{13}C NMR ($CDCl_3$) δ 157.3, 142.0, 140.3, 130.8, 129.6, 128.8, 126.8, 125.7, 120.3, 110.1, 55.1, 47.4, 46.1, 36.3, 32.5, 30.8, 30.2, 27.7, 27.0, 21.2, 19.9, 19.0, 13.5.

MS (ESI) $[M+H]^+$ calcd for $C_{18}H_{32}NO$: 278.2478, found: 278.2489.



(*R*)-*N*-(2-Ethyl-5-(4-fluorophenyl)pentyl)-*N*-(4-methoxybenzyl)-4-methylbenzenesulfonamide (Table 2, entry 1). *N*-(2-Bromobutyl)-*N*-(4-methoxybenzyl)-4-methylbenzenesulfonamide (213 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-4-fluorobenzene with 9-BBN dimer (1.5 M in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10%

acetonitrile/water→acetonitrile). Colorless oil. First run: 135 mg (56%, 89% ee). Second run: 143 mg (59%, 90% ee).

The ee was determined by HPLC on an AD-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 20.0 min (major), 26.1 min (minor).

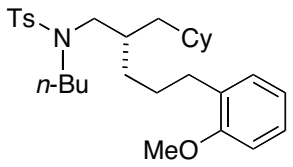
^{1}H NMR ($CDCl_3$) δ 7.68 (d, 2H, *J* = 8.4 Hz), 7.29 (d, 2H, *J* = 8.0 Hz), 7.13 (d, 2H, *J* = 8.4 Hz), 7.04–7.00 (m, 2H), 6.93–6.89 (m, 2H), 6.77 (d, 2H, *J* = 8.0 Hz), 4.21–4.11 (m, 2H), 3.74 (s, 3H), 2.91–2.89 (m, 2H), 2.42 (s, 3H), 2.39–2.35 (m, 2H), 1.40–1.07 (m, 7H), 0.63 (t, 3H, *J* = 7.6 Hz).

^{13}C NMR ($CDCl_3$) δ 159.1, 143.1, 136.7, 129.8, 129.63, 129.60, 129.5, 128.6, 127.3, 115.0, 114.8, 113.8, 55.2, 52.8, 52.6, 37.2, 35.3, 30.1, 28.3, 23.5, 21.5, 10.5.

FT-IR (film) 3020, 2965, 2811, 1700, 1653, 1558, 1540, 1507, 1457, 1215, 756, 669 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{28}H_{35}FNO_3S$: 484.2316, found: 484.2336.

$[\alpha]^{22}_D$ = +3.9 (c 1.0, $CHCl_3$) obtained with (*R,R*)-*m*-CF_3-DMPEDA.



(*S*)-*N*-Butyl-*N*-(2-(cyclohexylmethyl)-5-(2-methoxyphenyl)pentyl)-4-methylbenzenesulfonamide (Table 2, entry 2). *N*-(2-Bromo-3-cyclohexylpropyl)-*N*-butyl-4-methylbenzenesulfonamide (220 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 143 mg (56%, 90% ee). Second run: 131 mg (51%, 91% ee).

The ee was determined by HPLC on an AD-H column (1.0% *i*-PrOH in hexanes, 0.9 mL/min) with t_r = 15.3 min (major), 16.4 min (minor).

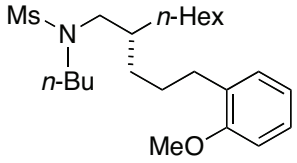
^{1}H NMR ($CDCl_3$) δ 7.67 (d, 2H, *J* = 8.4 Hz), 7.27 (d, 2H, *J* = 8.0 Hz), 7.18–7.10 (m, 2H), 6.89–6.82 (m, 2H), 3.80 (s, 3H), 3.11–2.87 (m, 4H), 2.56 (t, 2H, *J* = 7.6 Hz), 2.40 (s, 3H), 1.86–1.07 (m, 19H), 0.87 (t, 3H, *J* = 7.4 Hz), 0.77–0.74 (m, 3H).

^{13}C NMR ($CDCl_3$) δ 157.3, 142.8, 136.9, 130.9, 129.7, 129.4, 127.2, 126.8, 120.2, 110.1, 55.1, 52.9, 48.5, 39.5, 34.7, 33.9, 33.3, 33.0, 31.4, 30.5, 30.4, 26.6, 26.3, 26.2, 21.4, 20.0, 13.7.

FT-IR (film) 3546, 2927, 1600, 1494, 1464, 1340, 1243, 1159, 1091, 1051, 1030, 927, 815, 753, 656 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{30}H_{46}NO_3S$: 500.3193, found: 500.3185.

$[\alpha]^{24}_D$ = –6.5° (c 0.50, CH_2Cl_2) obtained with (*R,R*)-*m*-CF_3-DMPEDA.



(*R*)-*N*-Butyl-*N*-(2-(3-(2-methoxyphenyl)propyl)octyl)methanesulfonamide (Table 2, entry 3). *N*-(2-Bromooctyl)-*N*-butylmethanesulfonamide (171 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 158 mg (77%, 90% ee). Second run: 156 mg (76%, 90% ee).

The ee was determined by HPLC on an AS-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 10.5 min (minor), 18.1 min (major).

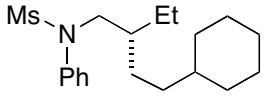
^{1}H NMR ($CDCl_3$) δ 7.17–7.09 (m, 2H), 6.88–6.81 (m, 2H), 3.79 (s, 3H), 3.12–2.99 (m, 4H), 2.77 (s, 3H), 2.57 (t, 2H, *J* = 7.6 Hz), 1.65–1.24 (m, 19H), 0.91 (t, 3H, *J* = 7.2 Hz), 0.86 (t, 3H, *J* = 6.8 Hz).

^{13}C NMR ($CDCl_3$) δ 157.3, 130.8, 129.7, 126.9, 120.3, 110.2, 55.2, 51.9, 48.0, 37.8, 36.1, 31.8, 31.0, 30.9, 30.6. 30.5, 29.6, 26.6, 26.2, 22.6, 20.1, 14.0, 13.7.

FT-IR (film) 3019, 2951, 2935, 1559, 1457, 1215, 760, 669 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{23}H_{42}NO_3S$: 412.2880, found: 412.2872.

$[\alpha]^{22}_D$ = –4.9 (c 1.0, $CHCl_3$) obtained with (*S*,*S*)-*m*-CF_3-DMPEDA.



(*R*)-*N*-(4-Cyclohexyl-2-ethylbutyl)-*N*-phenylmethanesulfonamide (Table 2, entry 4). *N*-(2-Bromobutyl)-*N*-phenylmethanesulfonamide (155 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of vinylcyclohexane with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). White solid. First run: 122 mg (71%, 71% ee). Second run: 111 mg (65%, 73% ee).

The ee was determined by HPLC on an OJ-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 8.2 min (major), 9.3 min (minor).

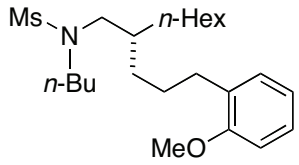
^{1}H NMR ($CDCl_3$) δ 7.40–7.28 (m, 5H), 3.57–3.48 (m, 2H), 2.82 (s, 3H), 1.64–1.61 (m, 5H), 1.41–0.98 (m, 12H), 0.78 (t, 3H, *J* = 7.2 Hz), 0.80–0.76 (m, 1H).

^{13}C NMR ($CDCl_3$) δ 139.4, 129.3, 128.2, 127.9, 53.7, 37.9, 37.6, 36.2, 33.7, 33.4, 33.3, 27.3, 26.7, 26.4, 26.3, 23.2, 10.2.

FT-IR (film) 2923, 2851, 1493, 1457, 1342, 1158, 1067, 964, 883, 775, 697 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{19}H_{32}NO_2S$: 338.2148, found: 338.2144.

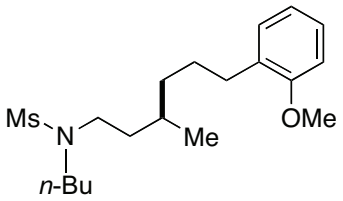
$[\alpha]^{24}_D$ = –19.4° (c 0.50, CH_2Cl_2) obtained with (*S*,*S*)-*m*-CF_3-DMPEDA.



(*R*)-*N*-Butyl-*N*-(2-(3-(2-methoxyphenyl)propyl)octyl)methanesulfonamide (eq 7). *N*-(2-Chlorooctyl)-*N*-butylmethanesulfonamide (149 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 148 mg (72%, 92% ee). Second run: 150 mg (73%, 91% ee).

The ee was determined by HPLC on an AS-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 9.0 min (minor), 15.5 min (major).

For characterization data, see Table 2, entry 3 (above).



(*R*)-*N*-Butyl-*N*-(6-(2-methoxyphenyl)-3-methylhexyl)methanesulfonamide (eq 8). *N*-(3-Bromobutyl)-*N*-butylmethanesulfonamide (150 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 149 mg (80%, 65% ee). Second run: 146 mg (78%, 62% ee).

The ee was determined by HPLC on an OJ-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 20.0 min (major), 24.6 min (minor).

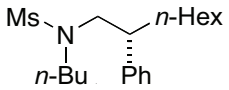
^{1}H NMR ($CDCl_3$) δ 7.15 (t, 1H, *J* = 7.8 Hz), 7.11 (d, 1H, *J* = 7.2 Hz), 6.88–6.82 (m, 2H), 3.80 (s, 3H), 3.22–3.11 (m, 4H), 2.78 (s, 3H), 2.63–2.52 (m, 2H), 1.67–1.17 (m, 11H), 0.95–0.90 (m, 6H).

^{13}C NMR ($CDCl_3$) δ 157.2, 130.8, 129.6, 126.7, 120.1, 110.1, 55.1, 47.2, 45.8, 38.1, 36.5, 35.5, 30.6, 30.4, 30.2, 27.0, 19.8, 19.3, 13.6.

FT-IR (film) 2932, 1601, 1588, 1494, 1464, 1377, 1331, 1243, 1147, 1098, 1051, 1032, 960, 926, 877, 754 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{19}H_{34}NO_3S$: 356.2254, found: 356.2243.

$[\alpha]^{24}_D$ = –8.1° (c 0.50, CH_2Cl_2) obtained with (*R*,*R*)-*m*-CF_3-DMPEDA.



(*R*)-*N*-Butyl-*N*-(2-phenyloctyl)methanesulfonamide (eq 9). *N*-(2-Bromooctyl)-*N*-butylmethanesulfonamide (170 mg, 0.50 mmol) and a solution of the distilled phenylborane prepared according to a literature procedure[4] (0.20 mL of Ph-(9-BBN) in 0.47 mL of *i*-Pr_2O) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 149 mg (88%, 96% ee). Second run: 151 mg (89%, 94% ee).

The ee was determined by HPLC on an AS-H column (2.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 15.4 min (major), 18.3 min (minor).

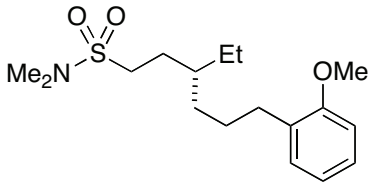
^{1}H NMR ($CDCl_3$) δ 7.30–7.27 (m, 2H), 7.21–7.17 (m, 3H), 3.38 (d, 2H, *J* = 7.2 Hz), 3.08–2.95 (m, 2H), 2.87–2.79 (m, 1H), 2.38 (s, 3H), 1.72–1.58 (m, 1H), 1.58–1.41 (m, 2H), 1.25–1.11 (m, 11H), 0.87 (t, 3H, *J* = 7.4 Hz), 0.81 (t, 3H, *J* = 6.8 Hz).

^{13}C NMR ($CDCl_3$) δ 142.8, 128.5, 128.0, 126.7, 53.3, 47.2, 44.8, 38.3, 33.3, 31.6, 29.9, 29.2, 27.2, 22.5, 19.8, 13.9, 13.6.

FT-IR (film) 3062, 3028, 2929, 2858, 1603, 1559, 1495, 1455, 1413, 1332, 1149, 1030, 962, 926, 783, 702, 518 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{19}H_{33}NNaO_2S$: 362.2124, found: 362.2137.

$[\alpha]^{24}_D$ = +15.2 (c 0.55, CH_2Cl_2) obtained with (*R*,*R*)-*m*-CF_3-DMPEDA.



(*R*)-3-Ethyl-6-(2-methoxyphenyl)-*N*,*N*-dimethylhexane-1-sulfonamide (eq 10). 3-Bromo-*N*,*N*-dimethylpentane-1-sulfonamide (150 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Colorless oil. First run: 148 mg (78%, 86% ee). Second run: 147 mg (77%, 84% ee).

The ee was determined by HPLC on an OD-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 16.3 min (major), 17.7 min (minor).

^{1}H NMR ($CDCl_3$) δ 7.15 (t, 1H, *J* = 7.6 Hz), 7.10 (d, 1H, *J* = 7.2 Hz), 6.88–6.81 (m, 2H), 3.80 (s, 3H), 2.84 (s, 8H), 2.58 (t, 2H, *J* = 7.6 Hz), 1.77–1.71 (m, 2H), 1.60–1.52 (m, 2H), 1.44–1.24 (m, 5H), 0.85 (t, 3H, *J* = 7.4 Hz).

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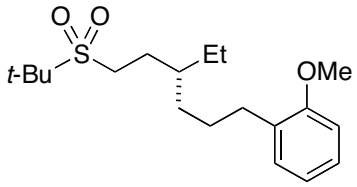
(4) Fang, G. Y.; Wallner, O. A.; Di Blasio, N.; Ginesta, X.; Harvey, J. N.; Aggarwal, V. K. *J. Am. Chem. Soc*. **2007**, *129*, 14632–14639.

^{13}C NMR ($CDCl_3$) δ 157.2, 130.5, 129.6, 126.8, 120.1, 110.1, 55.0, 45.8, 37.7, 37.3, 32.2, 30.2, 26.4, 25.8, 25.3, 10.5.

FT-IR (film) 2929, 1601, 1587, 1464, 1410, 1243, 1143, 1051, 1031, 960, 752, 703, 667 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{17}H_{30}NO_3S$: 328.1941, found: 328.1945.

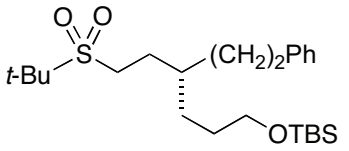
$[\alpha]^{25}_D = -9.6°$ (c 0.50, CH_2Cl_2) obtained with (*R*,*R*)-*m*-CF_3-DMPEDA.



(*R*)-1-(6-(*tert*-Butylsulfonyl)-4-ethylhexyl)-2-methoxybenzene (Table 3, entry 1). 1-(*tert*-Butylsulfonyl)-3-bromopentane (125 mg, 0.46 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.60 mL, 0.92 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Clear oil. First run: 122 mg (78%, 88% ee). Second run: 125 mg (80%, 88% ee).

The ee was determined by HPLC on an IA column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r: 25.5 min (major), 27.8 min (minor).

For characterization data, see eq 11 (below).



(*R*)-*tert*-Butyl(6-(*tert*-butylsulfonyl)-4-phenethylhexyloxy)dimethylsilane (Table 3, entry 2). (3-Bromo-5-(*tert*-butylsulfonyl)pentyl)benzene (175 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of allyloxy(*tert*-butyl)dimethylsilane with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.67 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Clear oil. First run: 184 mg (83%, 88% ee). Second run: 175 mg (79%, 86% ee).

The ee was determined by HPLC on an IB column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r: 11.6 min (minor), 12.8 min (major).

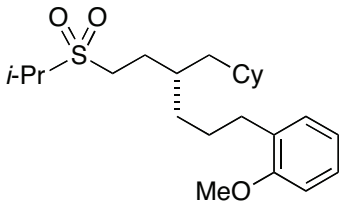
^{1}H NMR ($CDCl_3$) δ 7.27–7.23 (m, 2H), 7.15–7.14 (m, 3H), 3.58 (t, 2H, *J* = 5.8 Hz), 2.89–2.78 (m, 2H), 2.61 (t, 2H, *J* = 7.6 Hz), 1.96–1.82 (m, 2H), 1.67–1.48 (m, 5H), 1.38 (s, 11H), 0.87 (s, 9H), 0.03 (s, 6H).

^{13}C NMR ($CDCl_3$) δ 142.2, 128.3, 128.2, 125.7, 63.0, 58.8, 43.1, 36.1, 35.1, 32.7, 29.3, 28.9, 25.9, 23.7, 23.4, 18.2, –5.4.

FT-IR (film) 3062, 3027, 2933, 2857, 1604, 1496, 1462, 1388, 1363, 1293, 1256, 1116, 1007, 939, 836, 809, 776, 745, 700, 660 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{24}H_{44}NaO_3SSi$: 463.2673, found: 463.2660.

$[\alpha]^{24}_D$ = –2.5° (c 1.25, CH_2Cl_2) obtained with (*S*,*S*)-*m*-CF_3-DMPEDA.



(*S*)-1-(4-(Cyclohexylmethyl)-6-(isopropylsulfonyl)hexyl)-2-methoxybenzene (Table 3, entry 3). (2-Bromo-4-(isopropylsulfonyl)butyl)cyclohexane (165 mg, 0.51 mmol) and a solution of the alkylborane prepared by hydroboration of 2-methoxyallyl benzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.68 mL, 1.0 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Clear oil. First run: 170 mg (85%, 89% ee). Second run: 167 mg (84%, 90% ee).

The ee was determined by HPLC on an AD-H column (5.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r: 9.9 min (major), 11.2 min (minor).

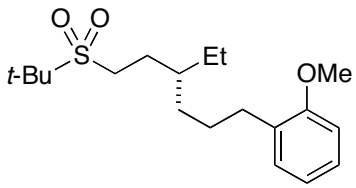
^1H NMR ($CDCl_3$) δ 7.15 (t, 1H, *J* = 7.6 Hz), 7.09 (d, 1H, *J* = 7.2 Hz), 6.87–6.81 (m, 2H), 3.80 (s, 3H), 3.07 (septet, 1H, *J* = 6.8 Hz), 2.82 (t, 2H, *J* = 8.2 Hz), 2.57 (t, 2H, *J* = 7.4 Hz), 1.77–1.52 (m, 10H), 1.42–0.99 (m, 14H), 0.85–0.77 (m, 2H).

^{13}C NMR ($CDCl_3$) δ 157.2, 130.5, 129.7, 126.9, 120.2, 110.1, 55.1, 52.2, 46.7, 41.5, 34.6, 33.54, 33.48, 33.1, 32.8, 30.3, 26.5, 26.2, 26.1, 24.9, 15.2, 15.1.

FT-IR (film) 2921, 1601, 1587, 1494, 1464, 1411, 1388, 1243, 1176, 1123, 1051, 1031, 967, 926, 880, 753, 683, 644, 565 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{23}H_{38}NaO_3S$: 417.2434, found: 417.2436.

$[\alpha]^{24}_D$ = +4.2° (c 0.5, CH_2Cl_2) obtained with (*R*,*R*)-*m*-CF_3-DMPEDA.



(*R*)-1-(6-(*tert*-Butylsulfonyl)-4-ethylhexyl)-2-methoxybenzene (eq 11). 1-(*tert*-Butylsulfonyl)-3-chloropentane (120 mg, 0.53 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-2-methoxybenzene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.70 mL, 1.1 mmol) were used. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile). Clear oil. First run: 138 mg (77%, 88% ee). Second run: 132 mg (73%, 88% ee).

The ee was determined by HPLC on an IA column (1.0% *i*-PrOH in hexanes, 1.0 mL/min) with t_r = 24.7 min (major), 27.1 min (minor).

^{1}H NMR ($CDCl_3$) δ 7.15 (t, 1H, *J* = 7.8 Hz), 7.10 (d, 1H, *J* = 7.2 Hz), 6.87–6.81 (m, 2H), 3.80 (s, 3H), 2.84–2.80 (m, 2H), 2.57 (t, 2H, *J* = 7.6 Hz), 1.86–1.80 (m, 2H), 1.57 (quintet, 2H, *J* = 7.8 Hz), 1.46–1.26 (m, 14H), 0.85 (t, 3H, *J* = 7.4 Hz).

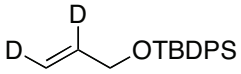
^{13}C NMR ($CDCl_3$) δ 157.4, 130.7, 129.7, 126.9, 120.3, 110.2, 58.9, 55.2, 43.4, 38.1, 32.4, 30.3, 26.5, 25.5, 23.5, 23.4, 10.6.

FT-IR (film) 2936, 1600, 1494, 1463, 1365, 1288, 1242, 1114, 1051, 1031, 806, 753, 657 cm^{-1}.

MS (ESI) $[M+Na]^+$ calcd for $C_{19}H_{32}NaO_3S$: 363.1964, found: 363.1985.

$[\alpha]^{24}_D$ = +0.31° (c 1.0, CH_2Cl_2) obtained with (*R*,*R*)-*m*-CF_3-DMPEDA.

IV. Transmetalation Study



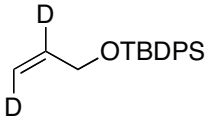
1-(*tert*-Butyldiphenylsiloxy)-2,3-*cis*-dideuterio-2-propene. Quinoline (1.6 mL, 14 mmol) was added to a flask that contained 5% Pd on $CaCO_3$ (12% w/w; 120 mg) and pentane (8.1 mL). The flask was evacuated and backfilled with argon three times. *tert*-Butyldiphenyl(prop-2-ynyloxy)silane (1.0 g, 3.4 mmol) was then added to the reaction mixture. The flask was evacuated and backfilled with D_2 three times. The reaction mixture was stirred for 45 min, and then it was filtered through celite. The filtrate was concentrated, and the residue was purified by chromatography (0%→50% Et_2O/hexanes), which furnished a colorless liquid (0.96 g, 95%).

^{1}H NMR ($CDCl_3$) δ 7.70 (d, 4H, *J* = 9.6 Hz), 7.45–7.36 (m, 6H), 5.37–5.36 (m, 1H), 4.22 (d, 2H, *J* = 1.2 Hz), 1.08 (s, 9H).

^{13}C NMR ($CDCl_3$) δ 136.6 (t, *J* = 22.5 Hz), 135.5, 133.7, 129.6, 127.6, 113.5 (t, *J* = 24.0 Hz), 64.5, 26.8, 19.3.

FT-IR (film) 3072, 2960, 2932, 2858, 1590, 1473, 1428, 1391, 1374, 1362, 1260, 1188, 1134, 1112, 1087, 1008, 938, 883, 823, 739, 615, 505 cm^{-1}.

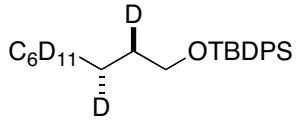
MS (ESI) $[M+Na]^+$ calcd for $C_{19}H_{22}D_2NaOSi$: 321.1614, found: 321.1634.



1-(*tert*-Butyldiphenylsiloxy)-2,3-*trans*-dideuterio-2-propene. The title compound was synthesized from (*E*)-2,3-dideuterioprop-2-en-1-ol[5] (1.0 g, 18 mmol), TBDPSCl (5.6 mL. 21

(5) Synthesized according to Baldwin, J. E.; Black, K. A. *J. Org. Chem.* **1983**, *48*, 2778–2779, using diethyl ether as solvent, with the addition of $LiAlD_4$ at 0 °C. After quenching and

mmol), and imidazole (2.7 g, 39 mmol) in DMF (13 mL), and purified by chromatography on reverse-phase silica (0%→100% acetonitrile/H_2O), which furnished a colorless oil (2.4 g; 46% over 2 steps). The ^{1}H NMR spectrum matched the reported data.[6]



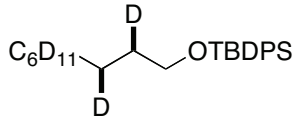
Eq 12. Bromocyclohexane-d_{11} (25 μL, 0.20 mmol) and a solution of the alkylborane prepared by hydroboration of 1-(*tert*-butyldiphenylsiloxy)-2,3-*trans*-dideuterio-2-propene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.30 mL, 0.40 mmol) were used, and the General Procedure (sulfonamides and sulfones) was followed. The product was purified by flash chromatography on silica gel (0%→50% Et_2O/hexane) and then again on reverse-phase silica gel (10% acetonitrile/water→acetonitrile), which furnished a colorless oil (51 mg, 64%).

^{1}H NMR (500 MHz, $CDCl_3$; deuterium decoupled) δ 7.63–7.62 (m, 4H), 7.39–7.32 (m, 6H), 3.58 (d, 2H, *J* = 7.0 Hz), 1.47 (br s, 1H), 1.11 (d, 1H *J* = 10.0 Hz), 1.00 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6, 134.2, 129.5, 127.5, 64.3, 32.7 (t, *J* = 19.5 Hz), 29.4 (t, *J* = 18.5 Hz), 26.9, 19.2.

FT-IR (film) 3071, 3050, 3000, 2930, 2858, 2197, 2102, 1590, 1472, 1428, 1389, 1361, 1112, 1008, 823, 739, 701, 613 cm^{-1}.

MS (ESI) $[M+H]^+$ calcd for $C_{25}H_{24}D_{13}OSi$: 394.3424, found: 394.3412.



Eq 13. Bromocyclohexane-d_{11} (25 μL, 0.20 mmol) and a solution of the alkylborane prepared by hydroboration of 1-(*tert*-butyldiphenylsiloxy)-2,3-*cis*-dideuterio-2-propene with 9-BBN dimer (1.5 M solution in *i*-Pr_2O; 0.30 mL, 0.40 mmol) were used, and the General Procedure (sulfonamides and sulfones) was followed. The product was purified by flash chromatography on reverse-phase silica gel (10% acetonitrile/water→acetonitrile), which furnished a colorless oil (64 mg, 80%).

^{1}H NMR (500 MHz, $CDCl_3$; deuterium decoupled) δ 7.66–7.65 (m, 4H), 7.38–7.36 (m, 6H), 3.61 (d, 2H, *J* = 6.5 Hz), 1.51 (br s, 1H), 1.14 (d, 1H *J* = 5.0 Hz), 1.03 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6, 134.2, 129.5, 127.6, 64.3, 32.8 (t, *J* = 19.5 Hz), 29.4 (t, *J* = 19.5 Hz), 26.9, 19.2.

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filtration over Celite, the solvent was removed at atmospheric pressure. Due to the low boiling point of the deuteriated alcohol, it was silylated immediately.

(6) Ridgway, B. H.; Woerpel, K. A. *J. Org. Chem.* **1998**, *63*, 458–460.

FT-IR (film) 3071, 3050, 3000, 2930, 2858, 2197, 2102, 1590, 1472, 1428, 1389, 1361, 1188, 1112, 1008, 823, 739, 701, 613 cm^{-1}.

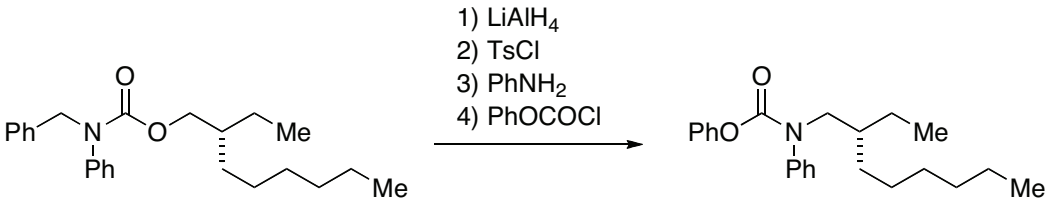
MS (ESI) $[M+H]^+$ calcd for $C_{25}H_{24}D_{13}OSi$: 394.3424, found: 394.3424.

V. Assignment of Absolute Stereochemistry

These procedures have not been optimized.

For one member of each family of directed reactions (three total), the absolute stereochemistry of the cross-coupling products was determined by correlation. The absolute stereochemistry of the other reaction products is assigned by analogy.

Absolute stereochemistry: carbamates.



prepared with (*R,R*) ligand according to:
Owston, N. A.; Fu, G. C.
J. Am. Chem. Soc. **2010**, *132*, 11908–11909.

(*R*)-Phenyl 2-ethyloctyl(phenyl)carbamate. (*R*)-2-Ethyloctyl benzyl(phenyl)carbamate was was obtained as described above.

$LiAlH_4$ (1.0 M in Et_2O; 0.65 mL, 0.64 mmol) was added to a solution of (*R*)-2-ethyloctyl benzyl(phenyl)carbamate (78 mg, 0.21 mmol) in THF (7.1 mL) in a 25-mL round-bottom flask equipped with a stir bar and a condenser under nitrogen. The reaction mixture was heated to 65 °C for 2 h, and then it was allowed to cool to r.t. Next, it was cooled to 0 °C, and the reaction was quenched with water (5 mL) and then aqueous HCl (1.0 M; 10 mL). The mixture was extracted with Et_2O (3 x 10 mL), and the organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes): 21 mg (64%).

Tosyl chloride (51 mg, 0.27 mmol) was added to a solution of the alcohol (21 mg, 0.13 mmol) in anhydrous pyridine (0.5 mL) in a 4-mL vial. The reaction mixture was stirred at r.t. for 24 h. Next, the reaction was quenched with H_2O (10 mL), and the mixture was extracted with Et_2O (3 x 10 mL). The organic extracts were washed with saturated aqueous $NaHCO_3$ (10 mL), dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes): 34 mg (83%).

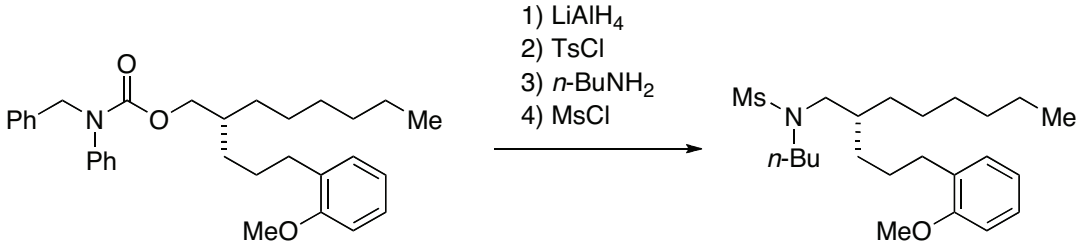
A 4-mL vial containing the tosylate (34 mg, 0.11 mmol), K_2CO_3 (30 mg, 0.22 mmol), NaI (0.027 mmol), and aniline (12 μL, 0.13 mmol) in MeCN (0.7 mL) was heated at 70 °C for 24 h. Next, the reaction mixture was allowed to cool to r.t., and the reaction was quenched with H_2O

(10 mL). The mixture was extracted with Et_2O (3 x 10 mL), and the organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes; not completely pure). The impure aniline was transferred to a 4-mL vial and dissolved in Et_2O (1.1 mL). Next, 2,6-lutidine (15 μL, 0.13 mmol) and then phenyl chloroformate (15 μL, 0.13 mmol) were added to the reaction mixture. The mixture was stirred at r.t. for 6 h, and then the reaction was quenched by the addition of HCl (1.0 M; 10 mL). The mixture was extracted with Et_2O (3 x 10 mL), and the organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes): 2 mg (5% over two steps).

HPLC conditions: AD-H column, 1.0% *i*-PrOH/hexanes, 1.0 mL/min. Retention times: 10.4 min (minor, *S*), 11.4 min (major, *R*).

The product from Table 1, entry 4, generated with (*R*,*R*)-DMPEDA: Retention times: 9.2 min (minor, *S*), 10.2 min (major, *R*).

Absolute stereochemistry: sulfonamides.



prepared with (*R*,*R*) ligand according to:
Owston, N. A.; Fu, G. C.
J. Am. Chem. Soc. **2010**, *132*, 11908–11909.

(*R*)-*N*-Butyl-*N*-(2-(3-(2-methoxyphenyl)propyl)octyl)methanesulfonamide. (*R*)-2-(3-(2-Methoxyphenyl)propyl)octyl benzyl(phenyl)carbamate was obtained as described above.

$LiAlH_4$ (1.0 M in Et_2O; 0.30 mL, 0.26 mmol) was added to a solution of (*R*)-2-(3-(2-methoxyphenyl)propyl)octyl benzyl(phenyl)carbamate (43 mg, 0.088 mmol) in THF (2.9 mL) in a 25-mL round-bottom flask equipped with a stir bar and a condenser under nitrogen. The reaction mixture was heated to 65 °C for 3.5 h, and then it was allowed to cool to r.t. Next, the reaction mixture was cooled to 0 °C and quenched with water (5 mL) and then aqueous HCl (1.0 M; 10 mL). The mixture was extracted with Et_2O (3 x 10 mL), and the organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes).

The alcohol was transferred to a 4-mL vial and dissolved in anhydrous pyridine (0.3 mL). Tosyl chloride (34 mg, 0.18 mmol) was added, and the reaction mixture was stirred at r.t. for 24 h. The reaction was quenched with H_2O (10 mL), and the mixture was extracted with Et_2O (3 x 10 mL). The organic extracts were washed with saturated aqueous $NaHCO_3$ (10 mL), dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes).

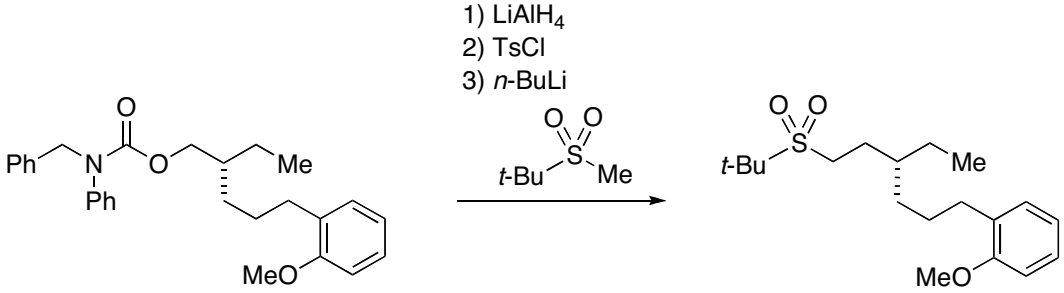
The tosylate, DMF (1.3 mL), *i*-Pr_2NEt (46 μL, 0.26 mmol), and *n*-$BuNH_2$ (87 μL, 0.88 mmol) were added to a 20-mL vial. The reaction mixture was heated at 60 °C for 48 h. Next, the reaction mixture was cooled to r.t., diluted with H_2O (10 mL), and extracted with Et_2O (3 x 10 mL). The organic extracts were washed with brine (4 x 10 mL), dried over $MgSO_4$, filtered, and concentrated by rotary evaporation.

The residue was transferred to a 20-mL vial and dissolved in CH_2Cl_2 (0.45 mL). Next, 2,6-lutidine (0.10 mL, 0.88 mmol) and mesyl chloride (70 μL, 0.88 mmol) were added sequentially. The reaction was stirred at r.t. for 7.5 h. Then, the reaction was quenched with aqueous HCl (1.0 M; 10 mL), and the mixture was extracted with Et_2O (3 x 10 mL). The organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes): 19 mg (53% over four steps).

HPLC conditions: AS-H column, 1.0% *i*-PrOH/hexanes, 0.8 mL/min. Retention times: 12.3 min (minor, *S*), 18.3 min (major, *R*).

The product from Table 2, entry 3, generated with (*S,S*)-*m*-CF_3-DMPEDA: Retention times: 12.2 min (major, *R*), 18.4 min (minor, *R*).

Absolute stereochemistry: sulfones.



prepared with (*R,R*) ligand according to:
Owston, N. A.; Fu, G. C.
J. Am. Chem. Soc. **2010**, *132*, 11908–11909.

(*R*)-1-(6-(*tert*-Butylsulfonyl)-4-ethylhexyl)-2-methoxybenzene. (*R*)-2-Ethyl-5-(2-methoxyphenyl)pentyl benzyl(phenyl)carbamate was obtained as described above.

$LiAlH_4$ (1.0 M in Et_2O; 1.3 mL, 1.3 mmol) was added to a solution of (*R*)-2-ethyl-5-(2-methoxyphenyl)pentyl benzyl(phenyl)carbamate (186 mg, 0.43 mmol) in THF (14.4 mL) in a 50-mL round-bottom flask equipped with a stir bar and a condenser under nitrogen. The reaction mixture was heated to 65 °C for 4.5 h, and then it was allowed to cool to r.t. The mixture was then cooled to 0 °C, and the reaction was quenched by the addition of water (5 mL) and then aqueous HCl (1.0 M; 10 mL). The mixture was extracted with Et_2O (3 x 10 mL), and the combined organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes): 79 mg (82%).

The alcohol (79 mg, 0.36 mmol) was transferred to a 4-mL vial and dissolved in anhydrous pyridine (1 mL). Tosyl chloride (137 mg, 0.72 mmol) was added, and the reaction mixture was

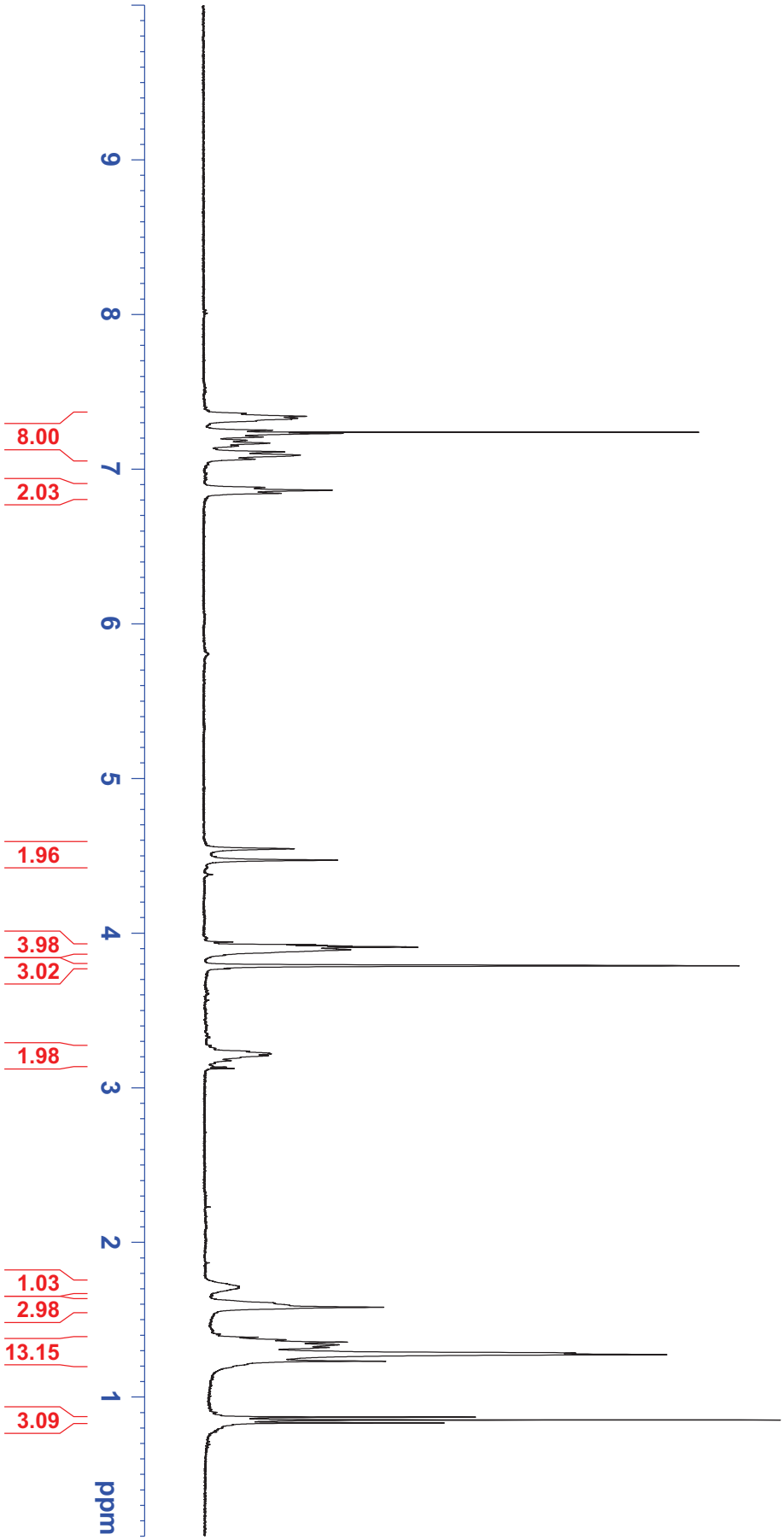
stirred at r.t. for 12 h. The reaction was quenched by the addition of H_2O (10 mL), and the mixture was extracted with Et_2O (3 x 10 mL). The organic extracts were washed with saturated aqueous $NaHCO_3$ (10 mL), dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes): 126 mg (93%).

n-BuLi (2.3 M, 0.15 mL) was added to a 20-mL vial containing a solution of 2-methyl-2-(methylsulfonyl)propane (46 mg, 0.34 mmol) in THF (2.3 mL) at –20 °C. The reaction mixture was allowed to warm to r.t. and then stirred for 30 min. The tosylate (126 mg, 0.33 mmol) was dissolved in THF (0.3 mL) and added to the reaction mixture at r.t. After 24 h, the reaction was quenched with H_2O (10 mL), and the mixture was extracted with Et_2O (3 x 10 mL). The organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. The residue was purified by flash chromatography (5%→100% Et_2O/hexanes): 75 mg (65%).

HPLC conditions: IA column, 1.0% *i*-PrOH/hexanes, 1.0 mL/min. Retention times: 20.0 min (major, *R*), 22.0 min (minor, *S*).

The product from Table 3, entry 1, generated with (*R*,*R*)-*m*-CF_3-DMPEDA: Retention times: 20.0 min (major, *R*), 21.1 min (minor, *S*).

VI. ^{1}H NMR Spectra



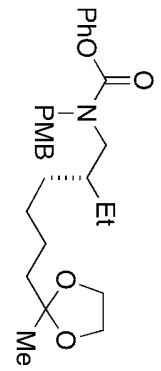


Table 1, entry 1

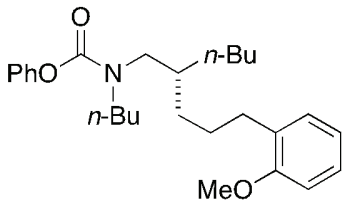
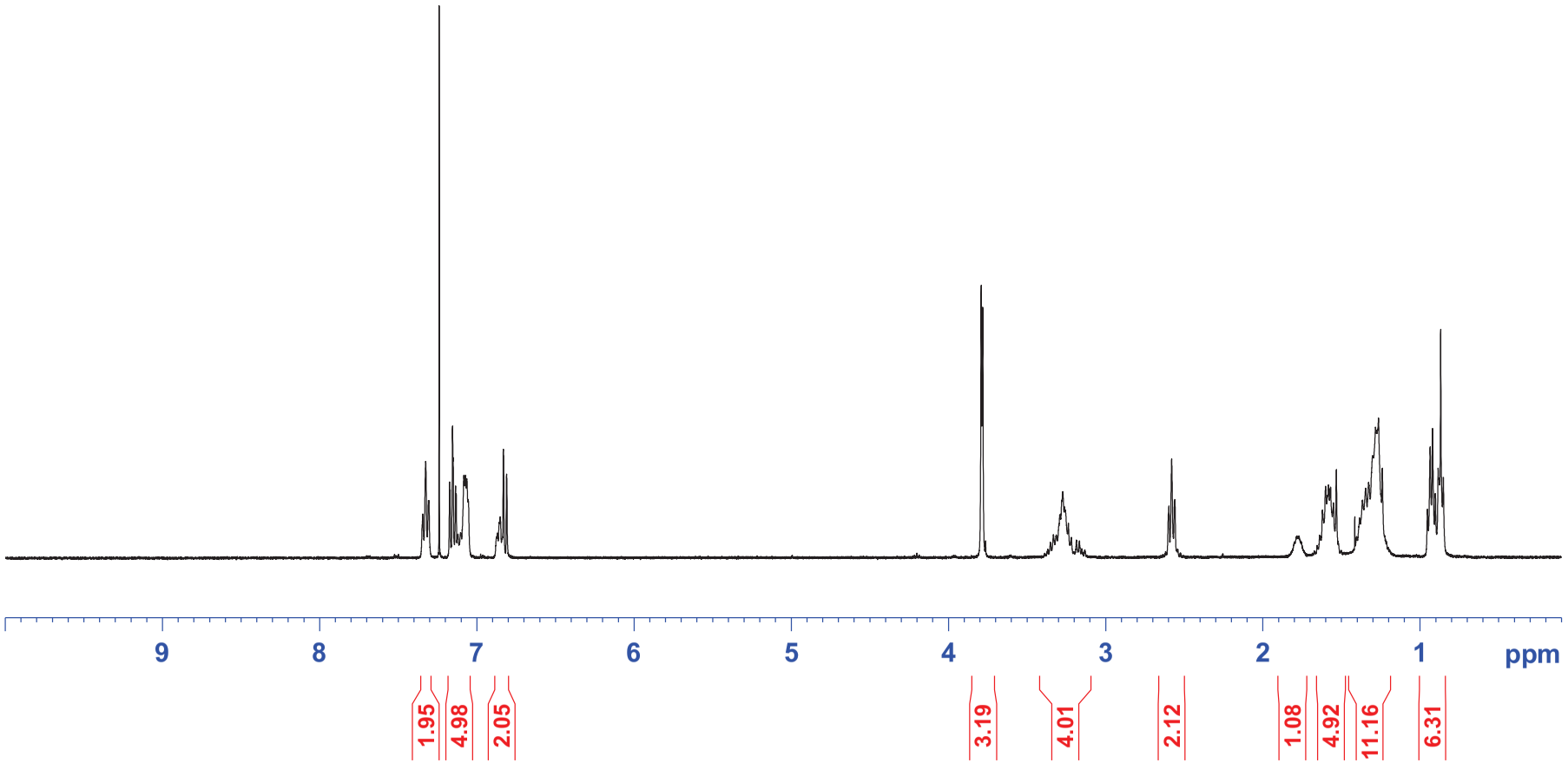


Table 1, entry 2



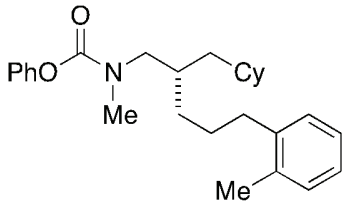
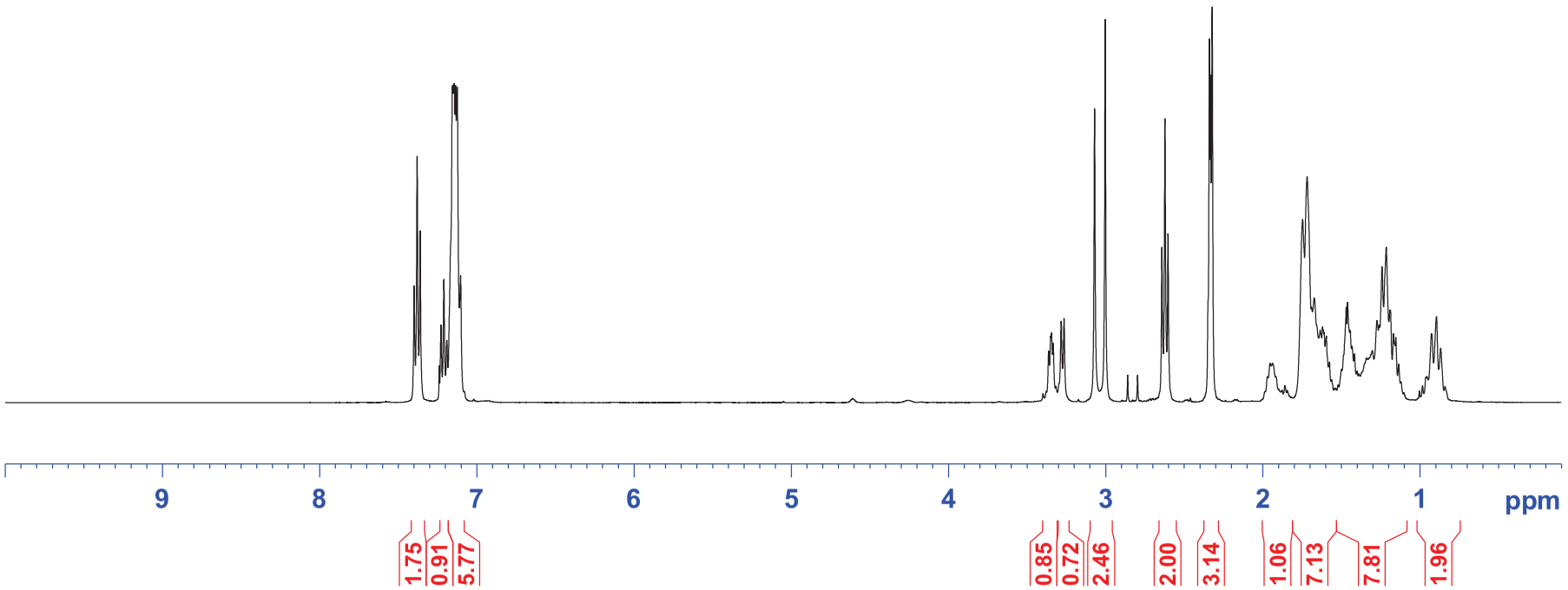


Table 1, entry 3



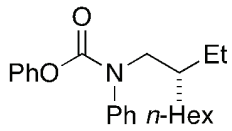
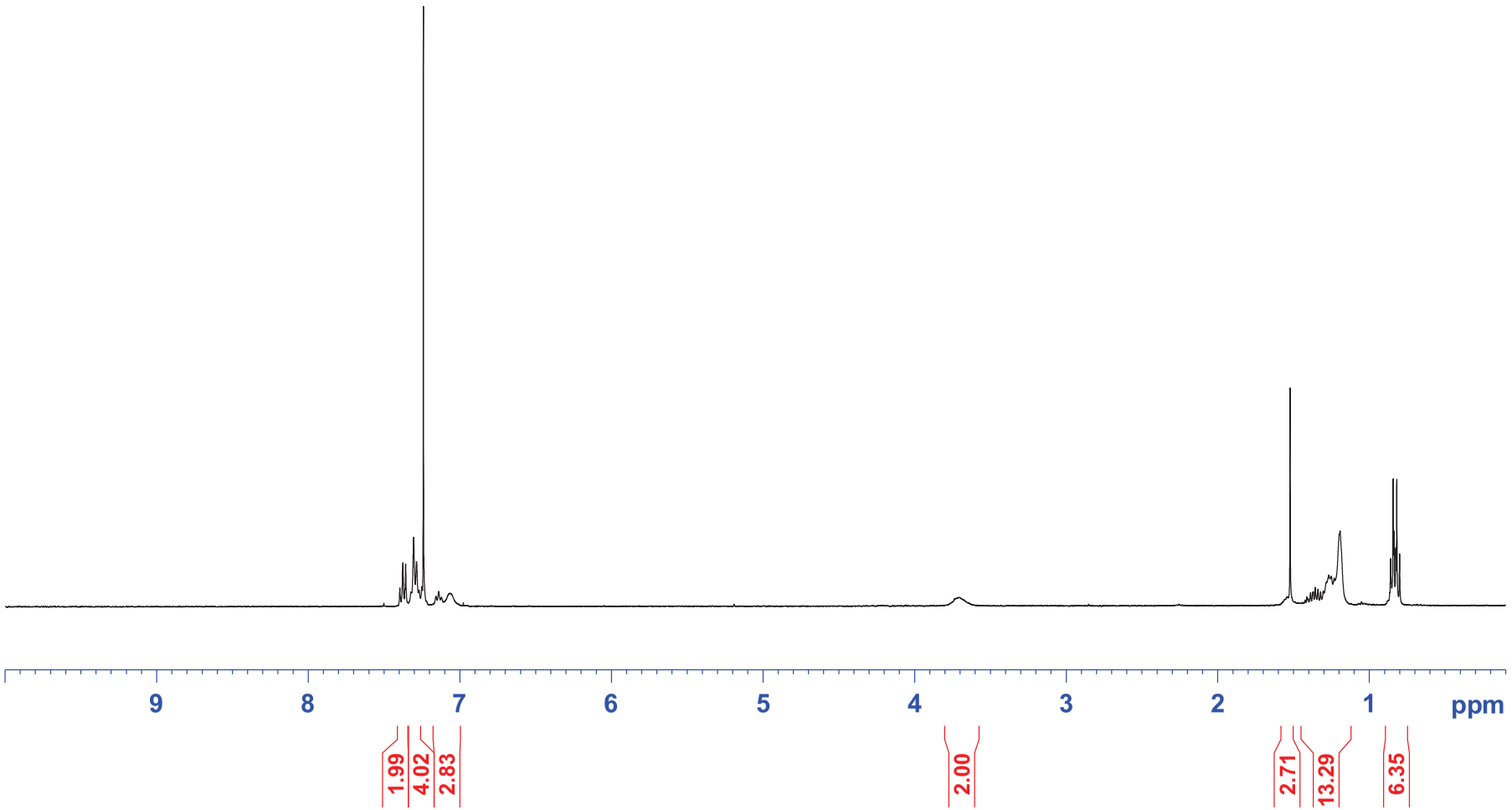


Table 1, entry 4



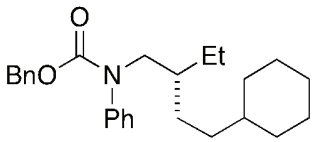
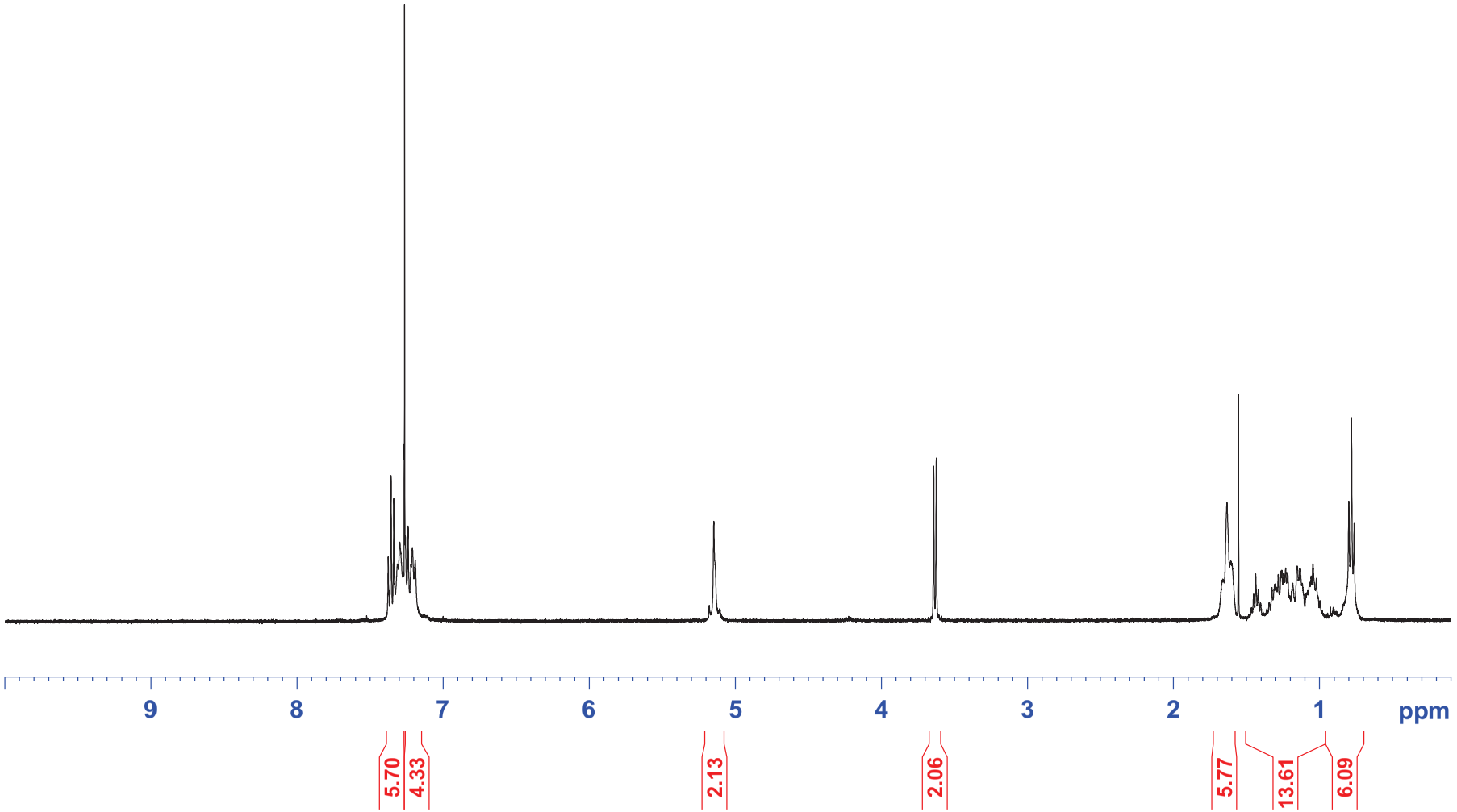
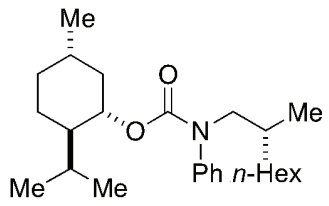


Table 1, entry 5

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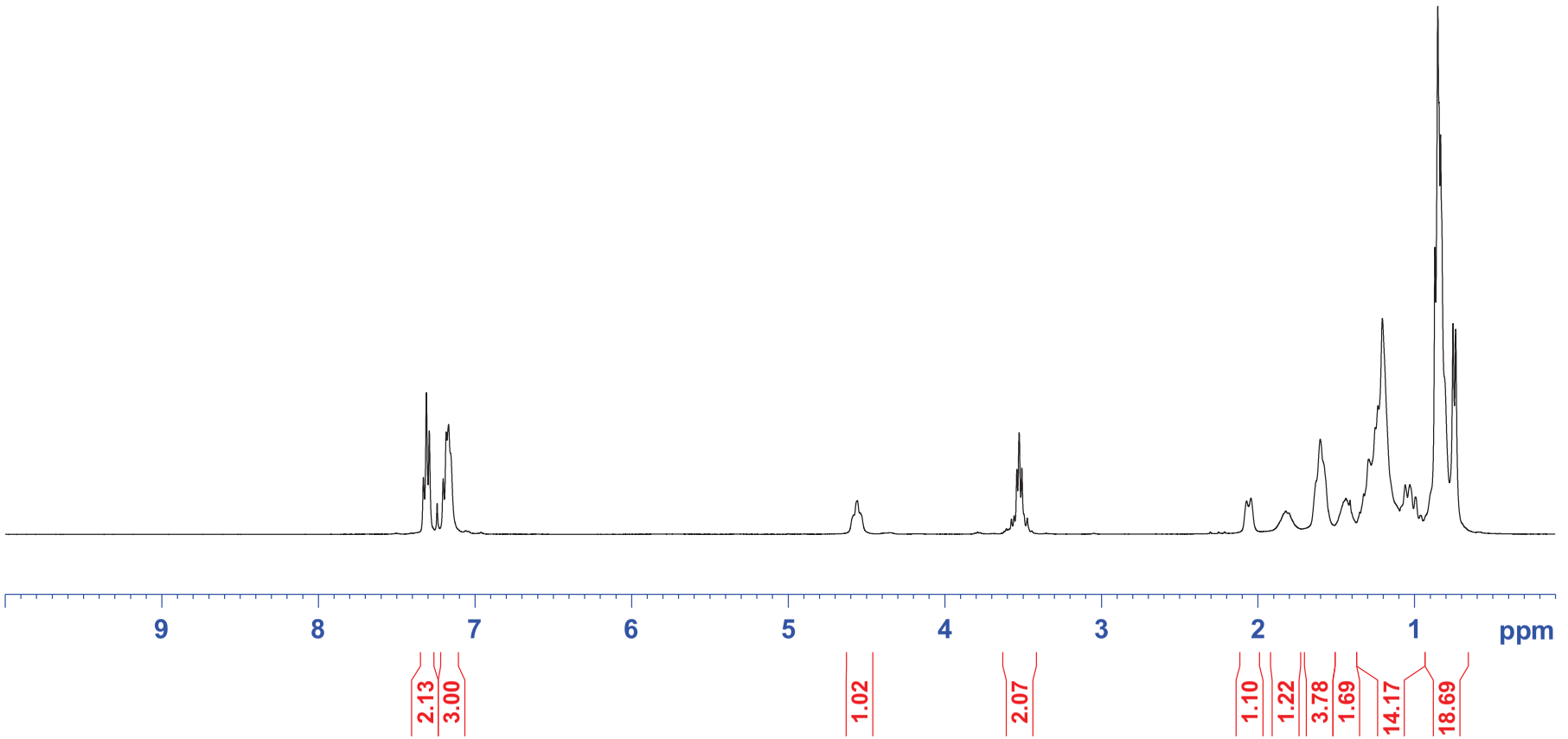
N
Me
Ph n-Hex
Me
Me

eq 2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2.07
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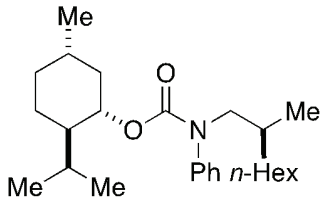
3.78
1.69
14.17
18.69


Me
O
O
N
Me
Ph n-Hex
Me
Me

eq 3

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7
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4
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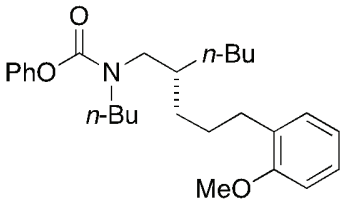
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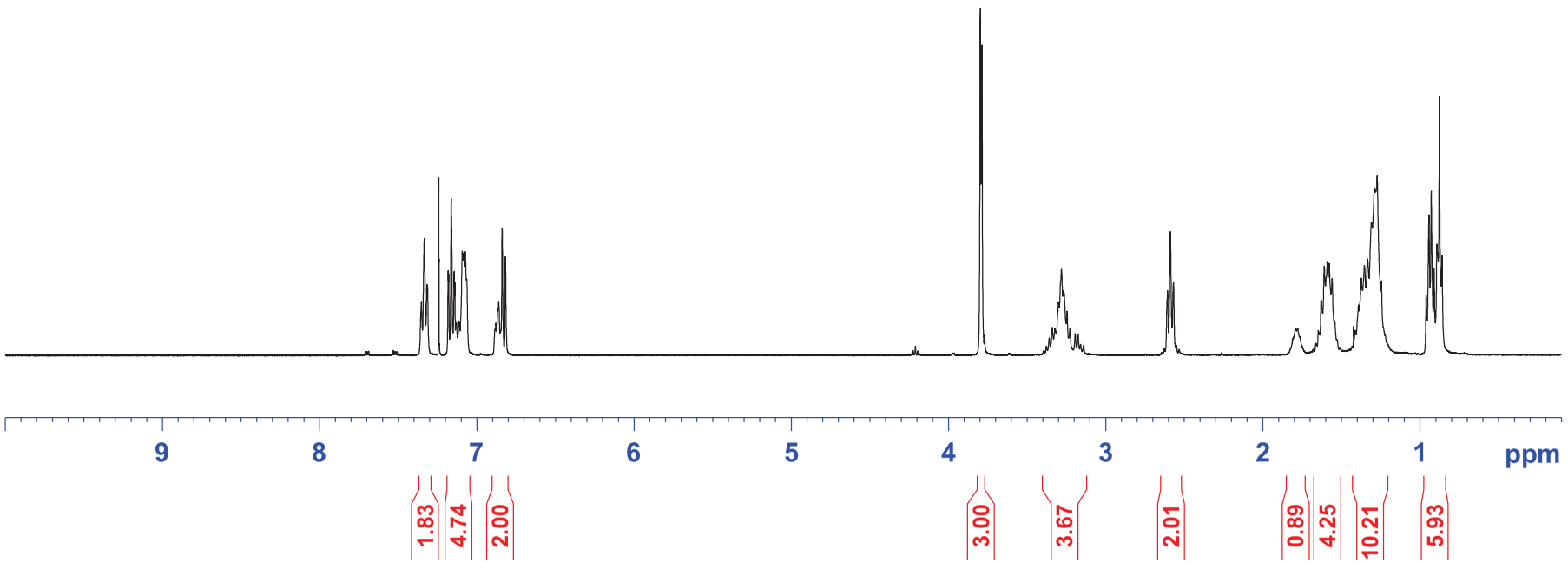
n-Bu
MeO

eq 4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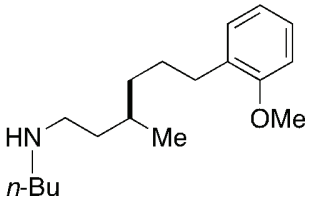
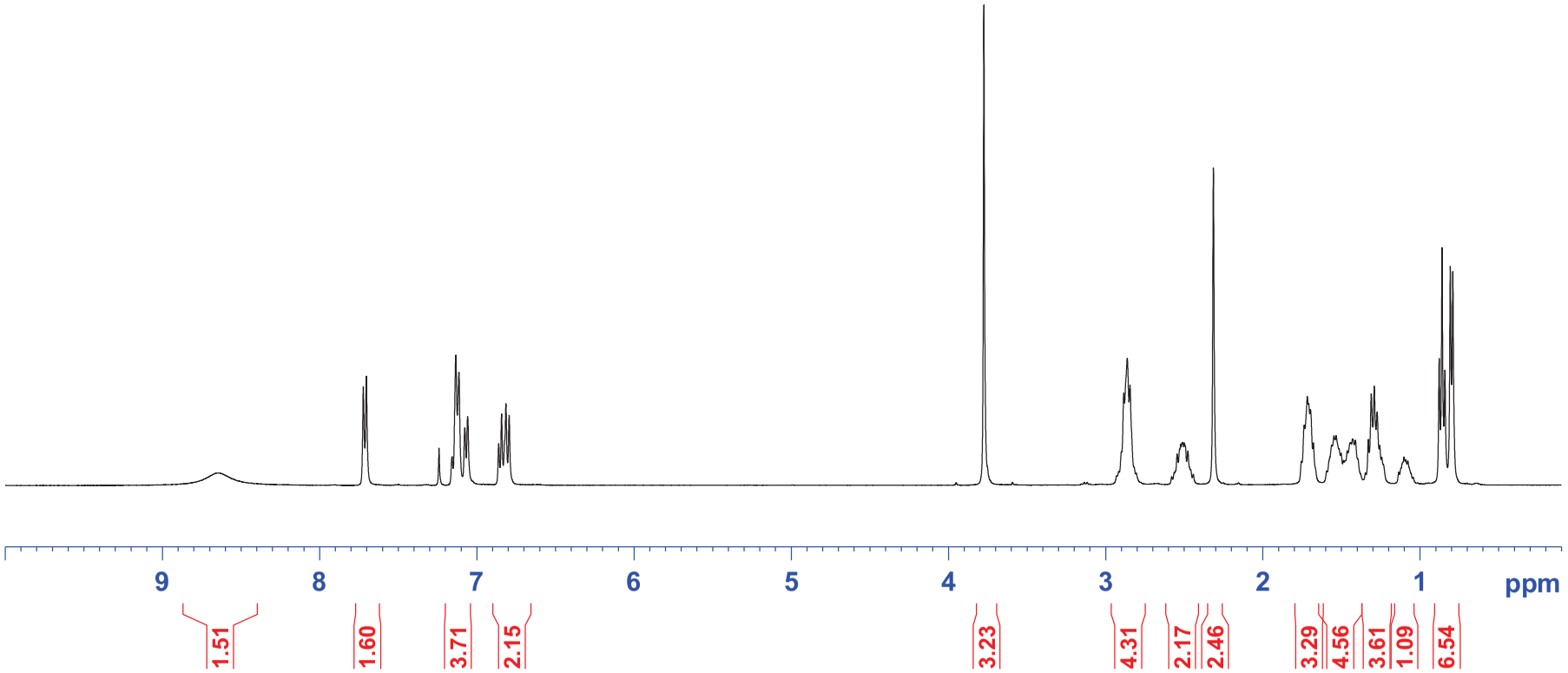
eq 5


HN
n-Bu
Me
OMe

eq 6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1.09
6.54

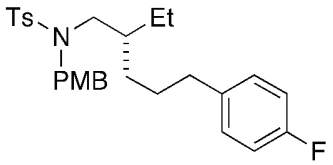
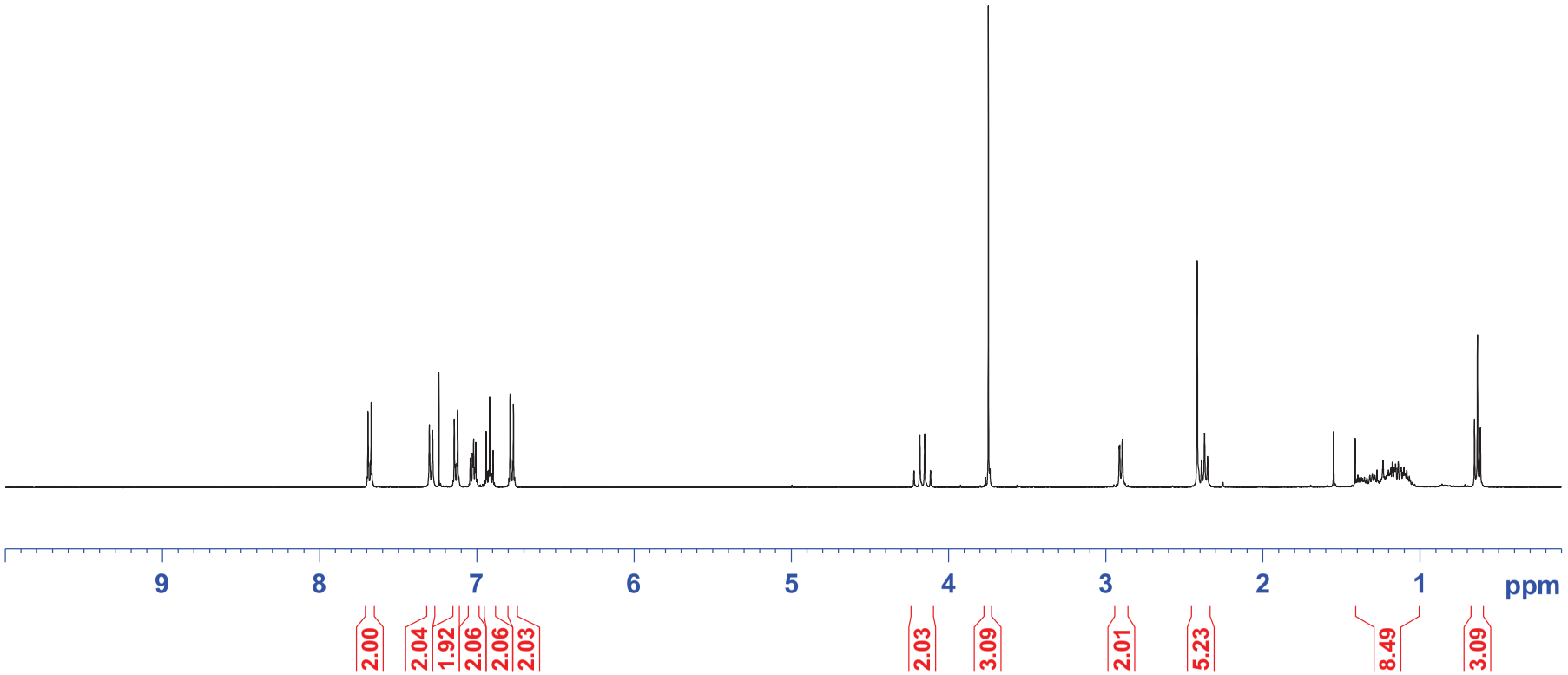
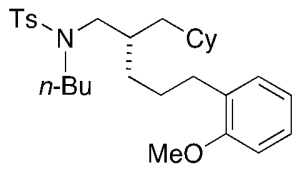


Table 2, entry 1





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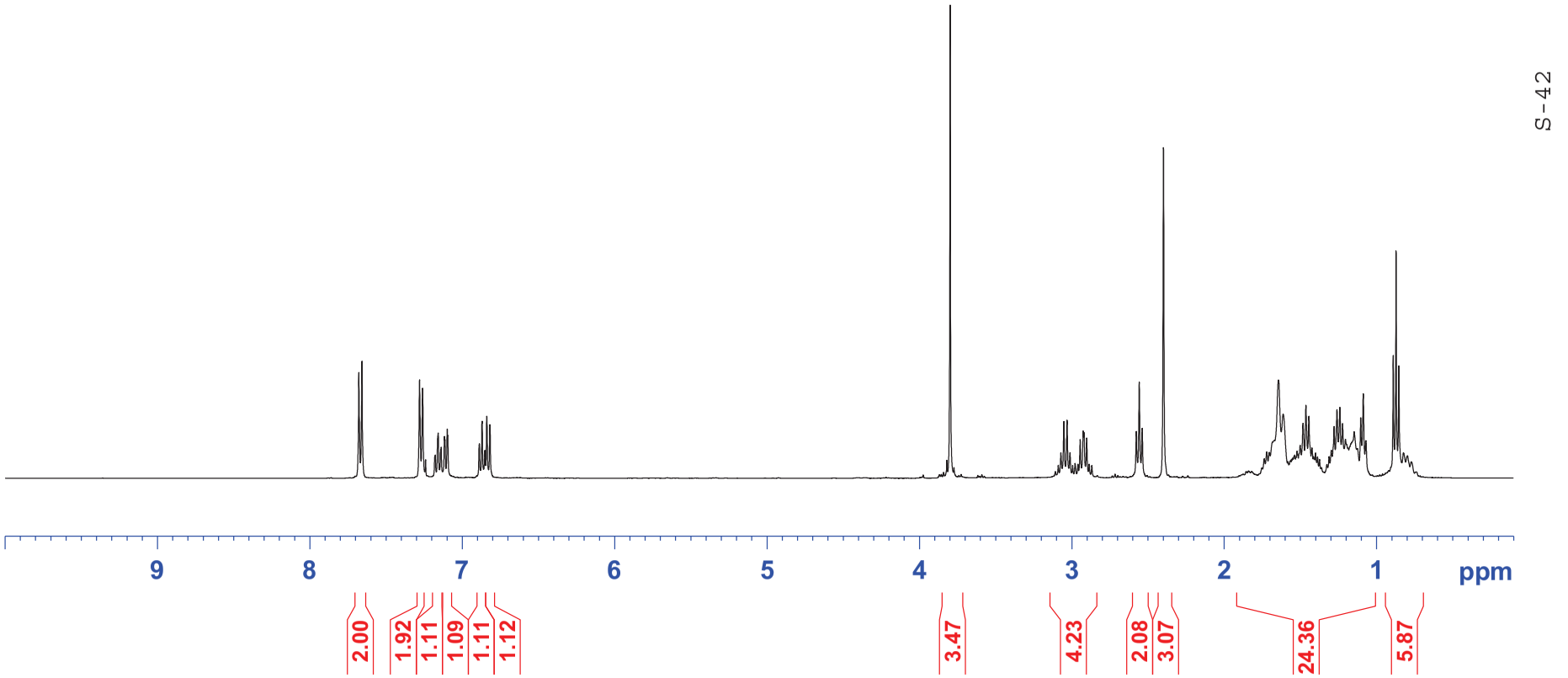

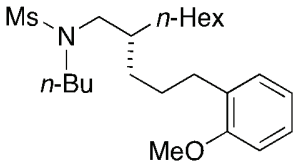

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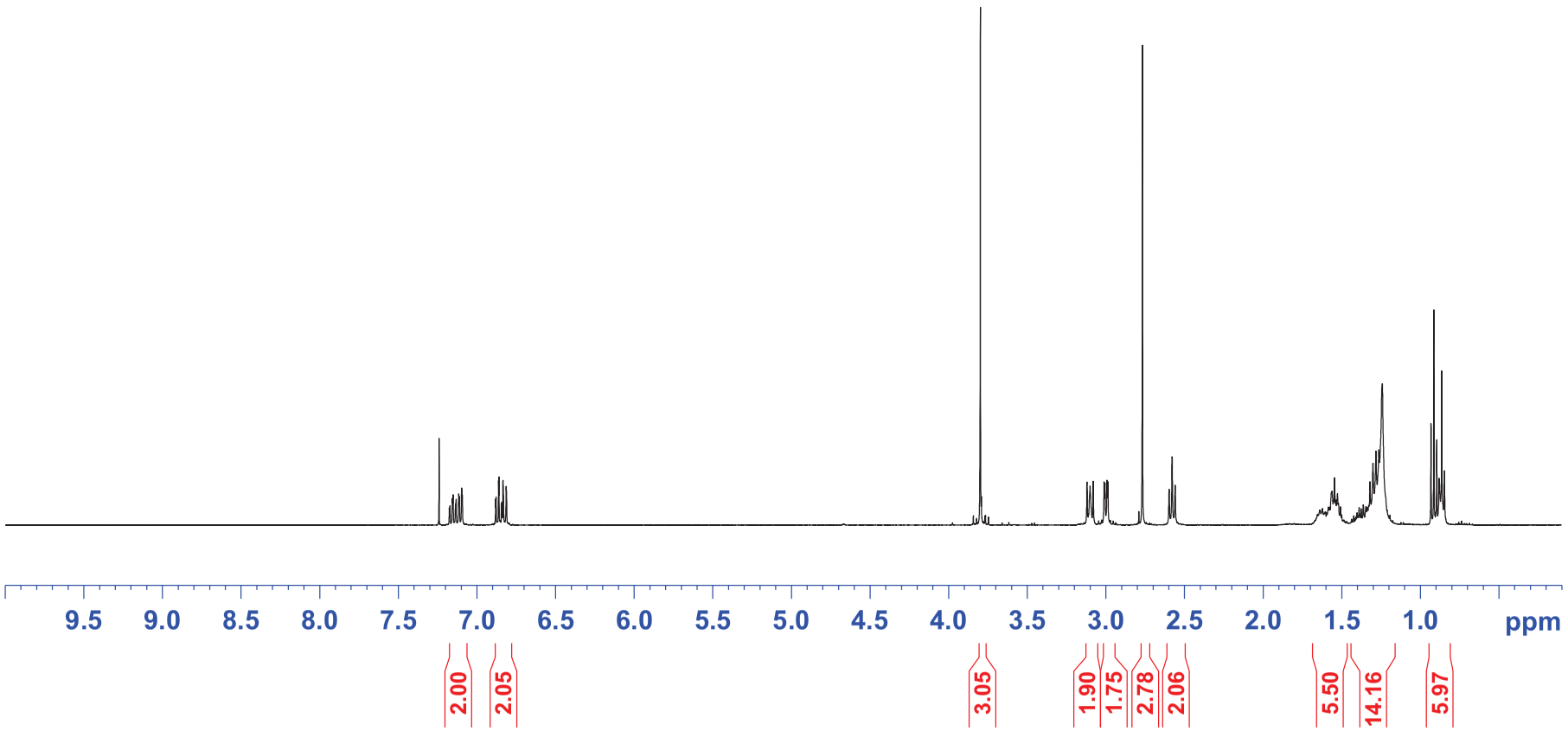


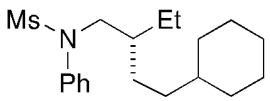
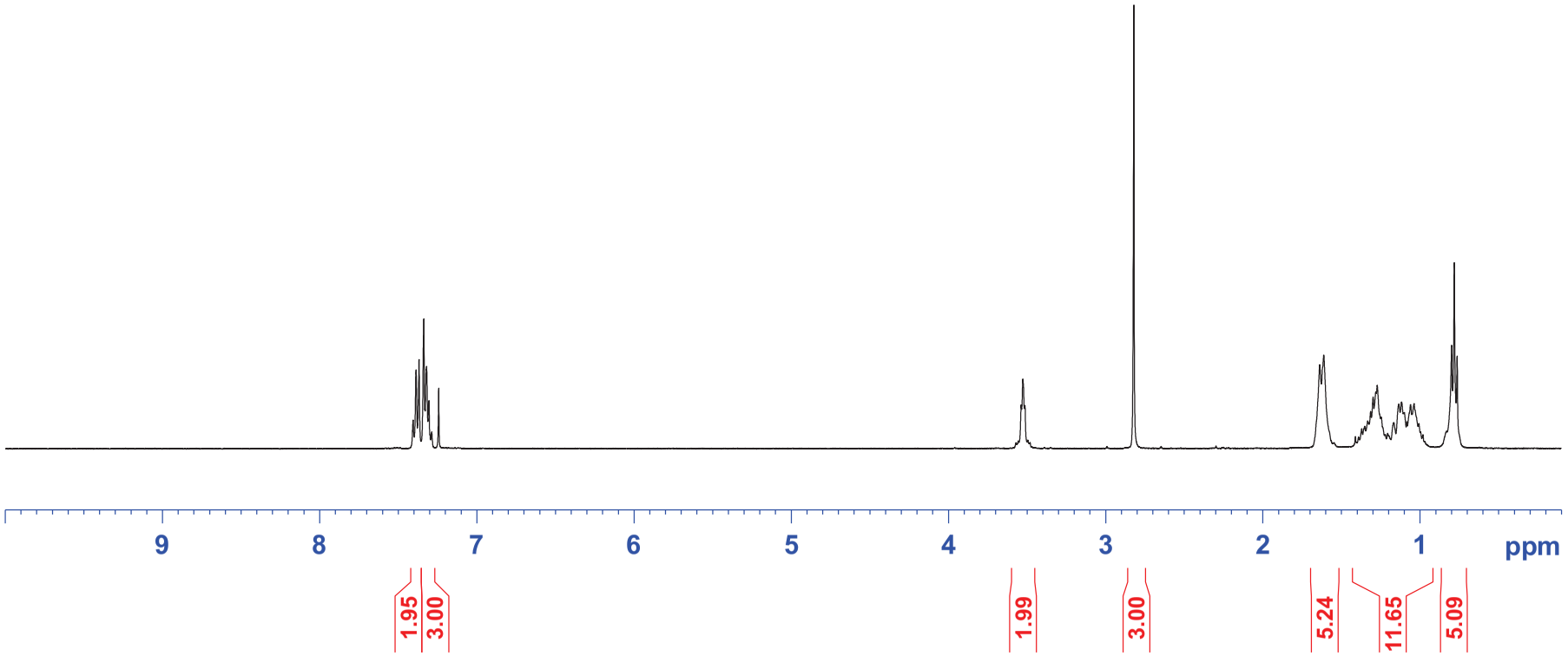
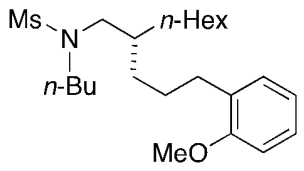


Table 2, entry 4

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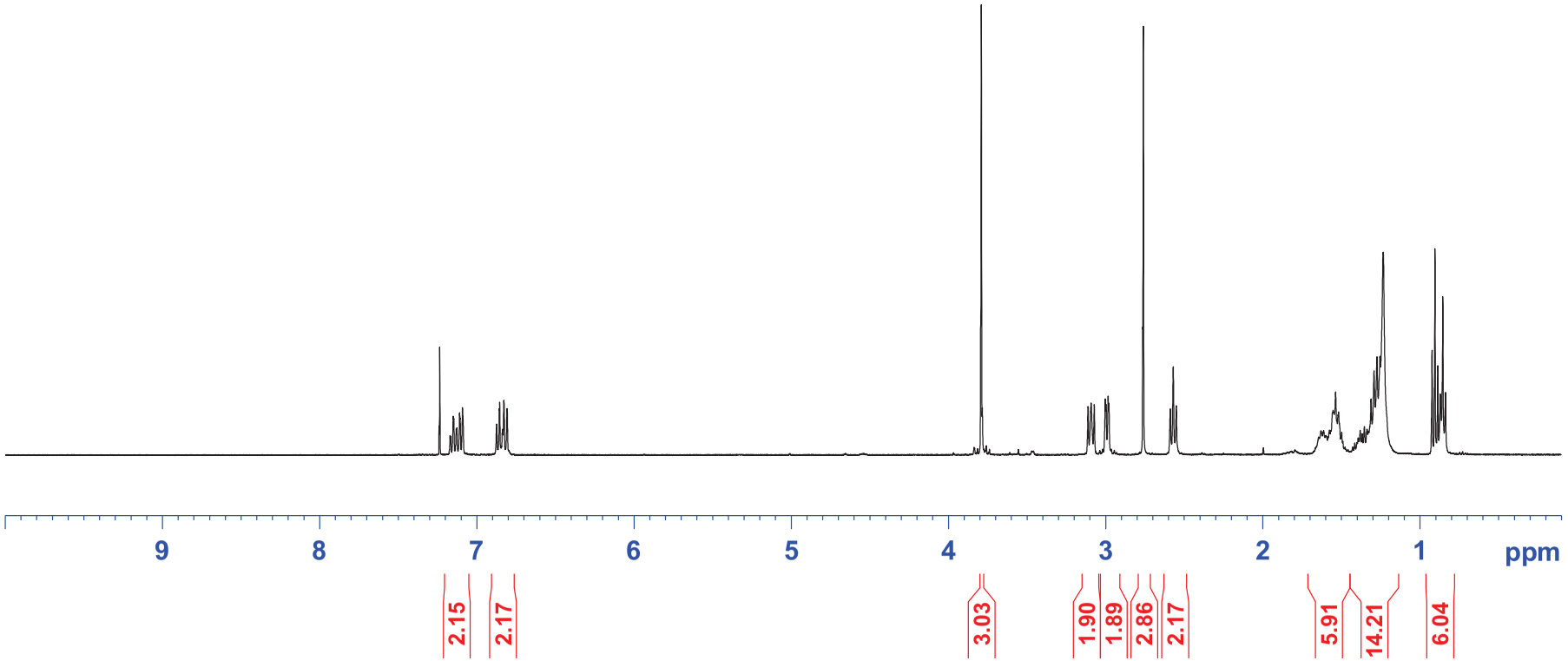
n-Hex
n-Bu
MeO

eq 7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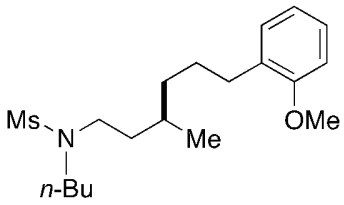
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n-Bu
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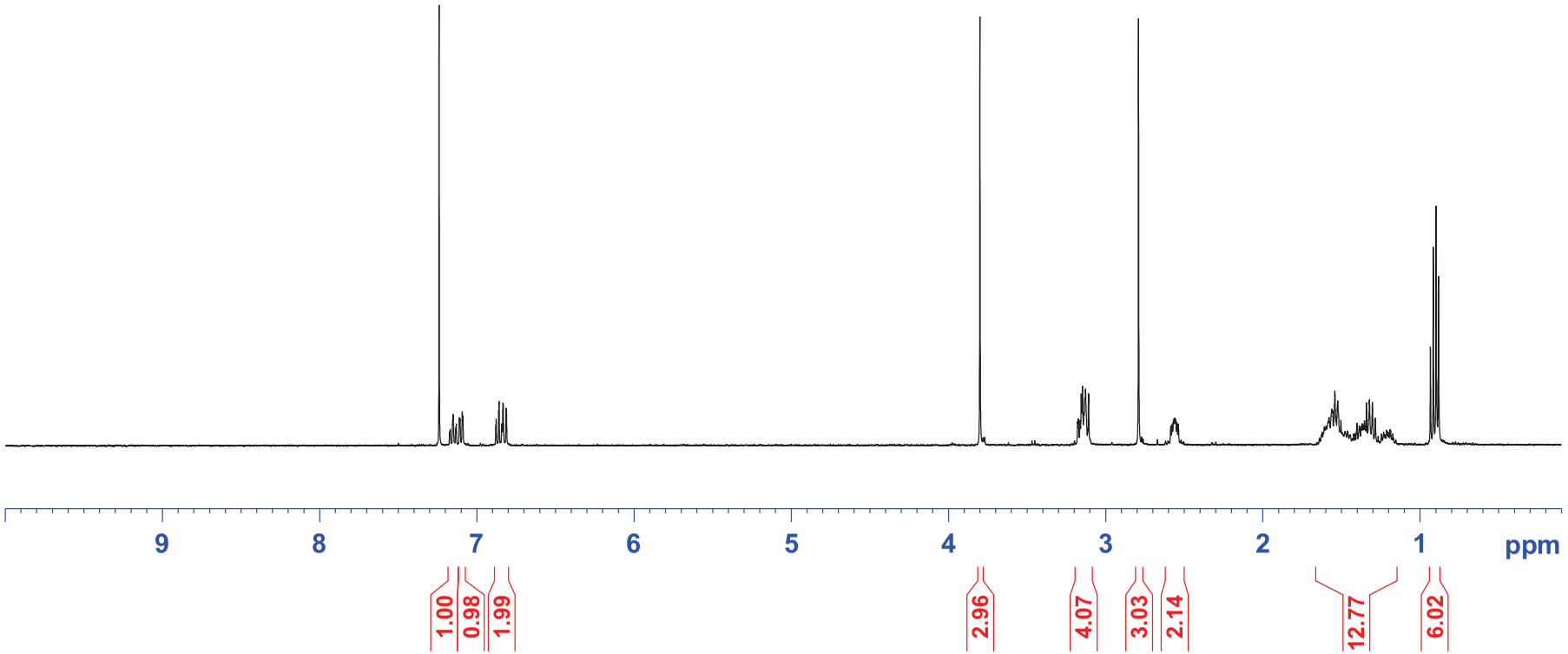
OMe

eq 8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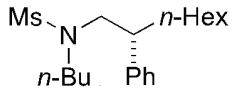
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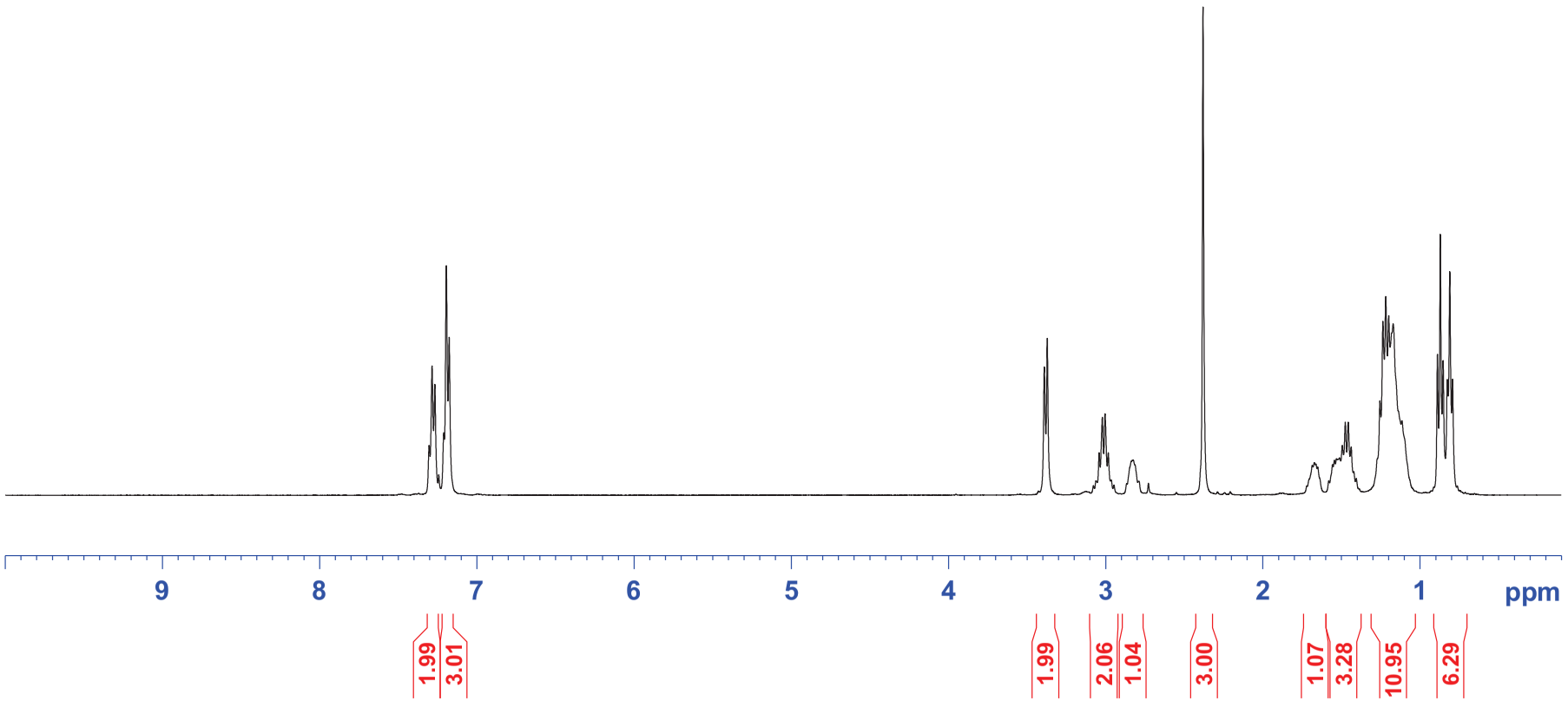
Ph

eq 9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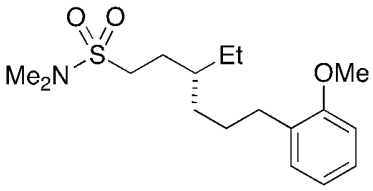
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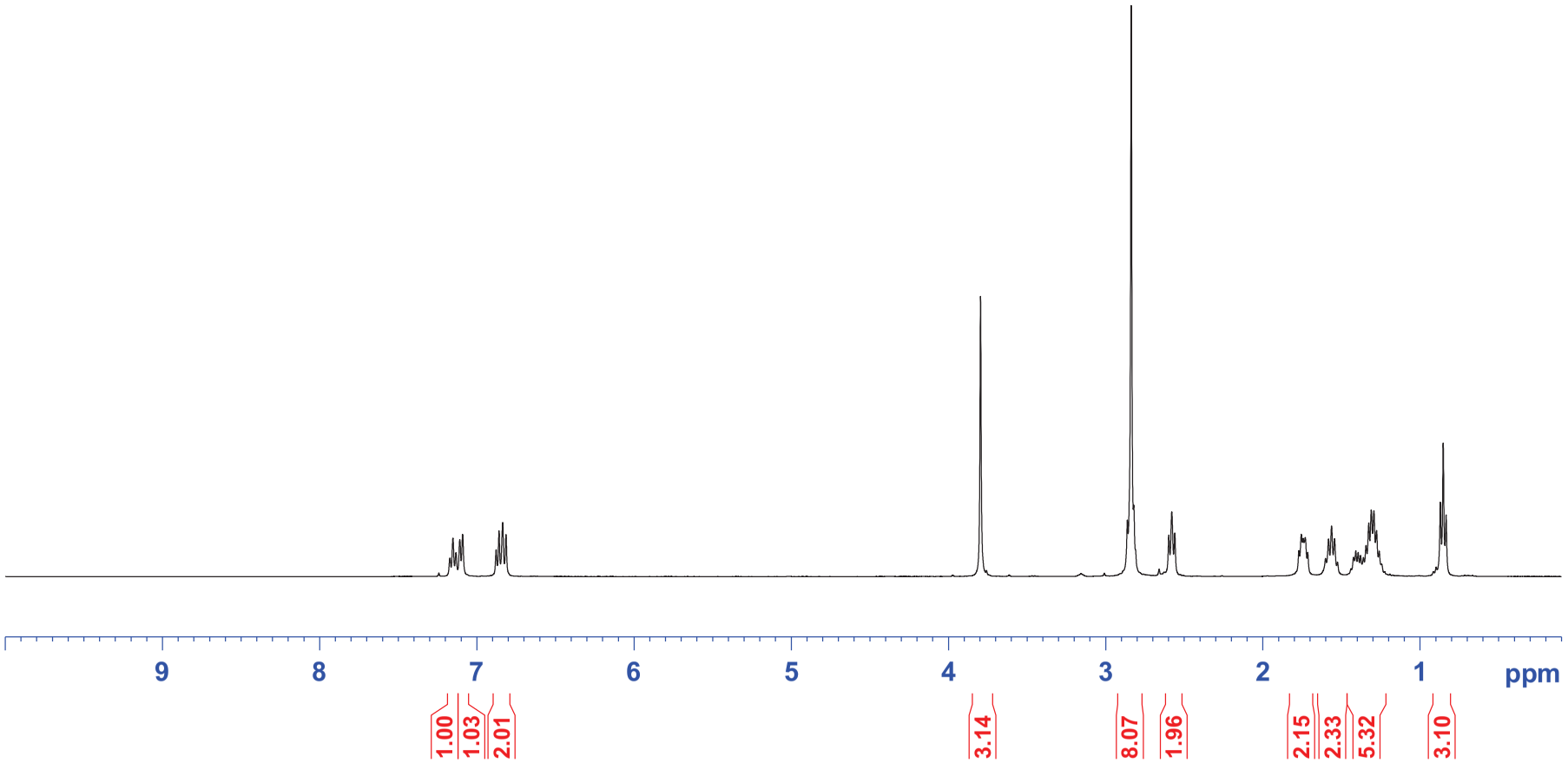
S
Me2N
Et
OMe

eq 10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3.10

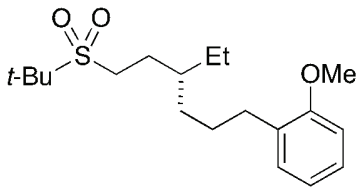
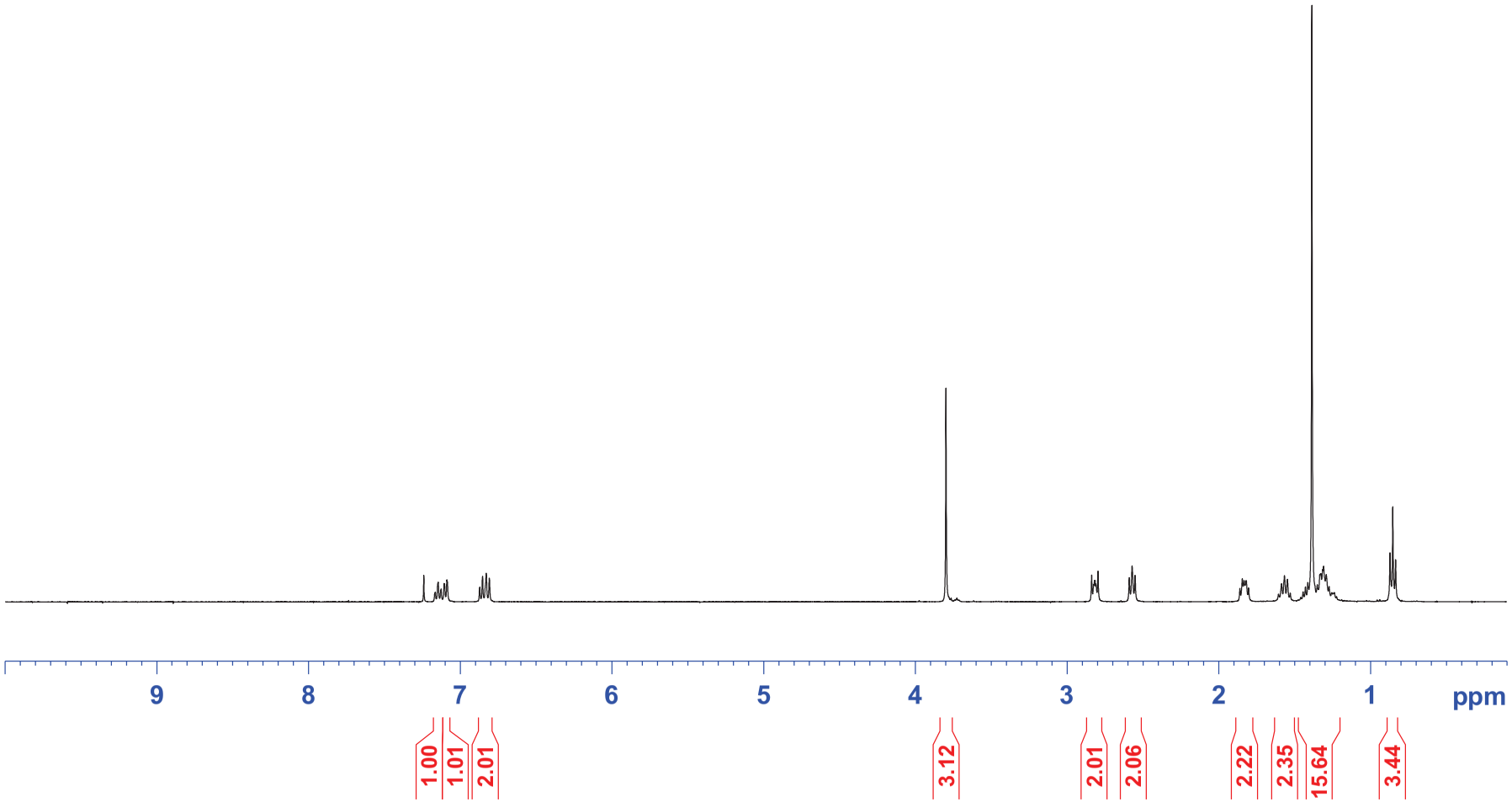


Table 3, entry 1



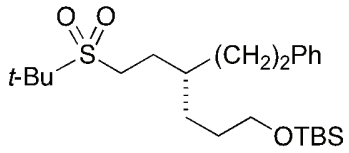
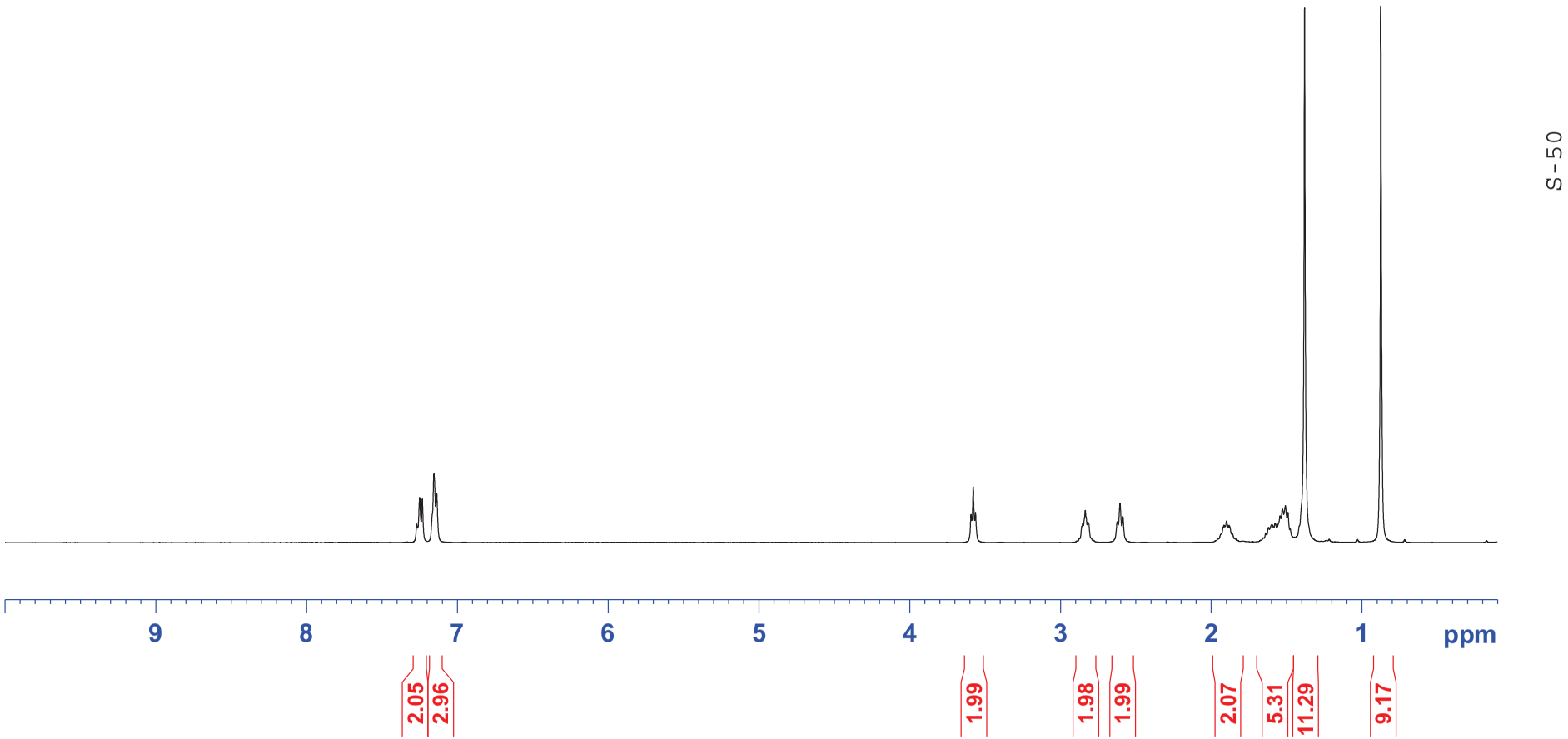


Table 3, entry 2



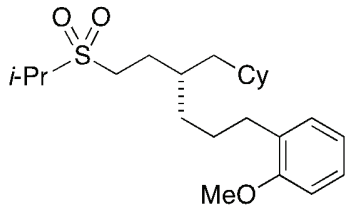
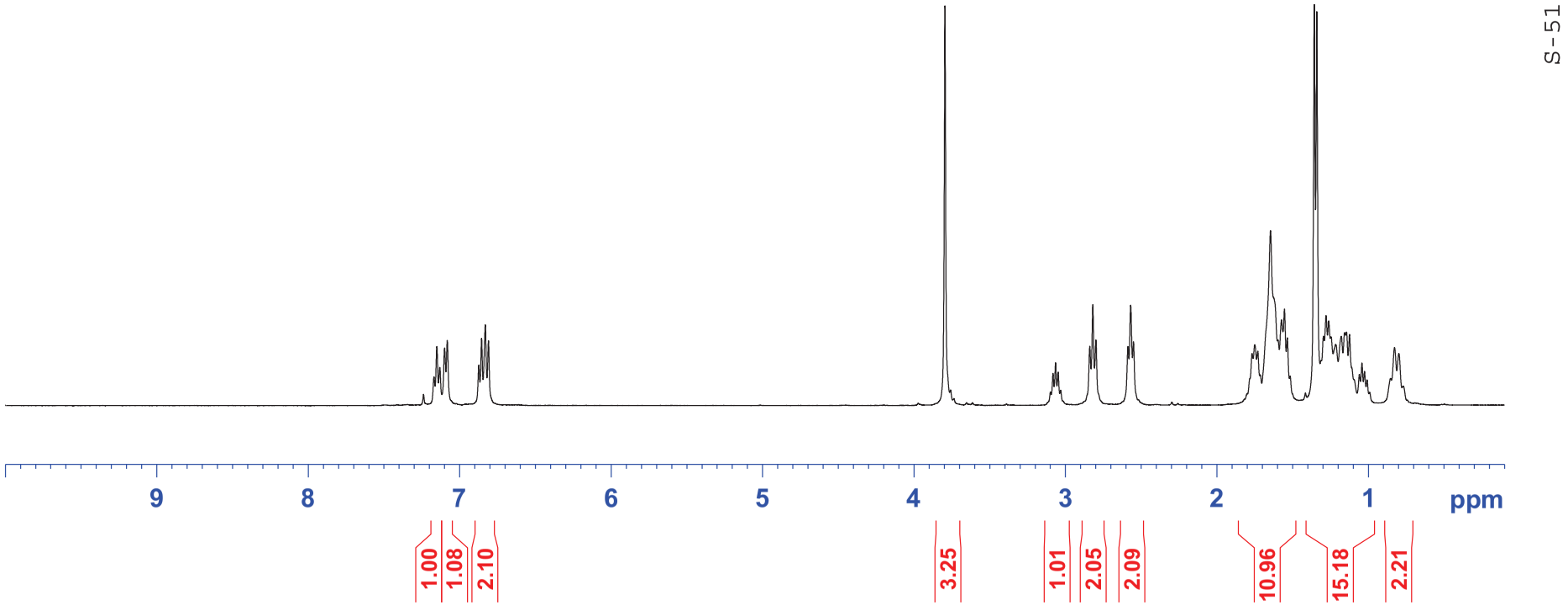
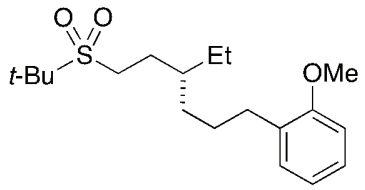
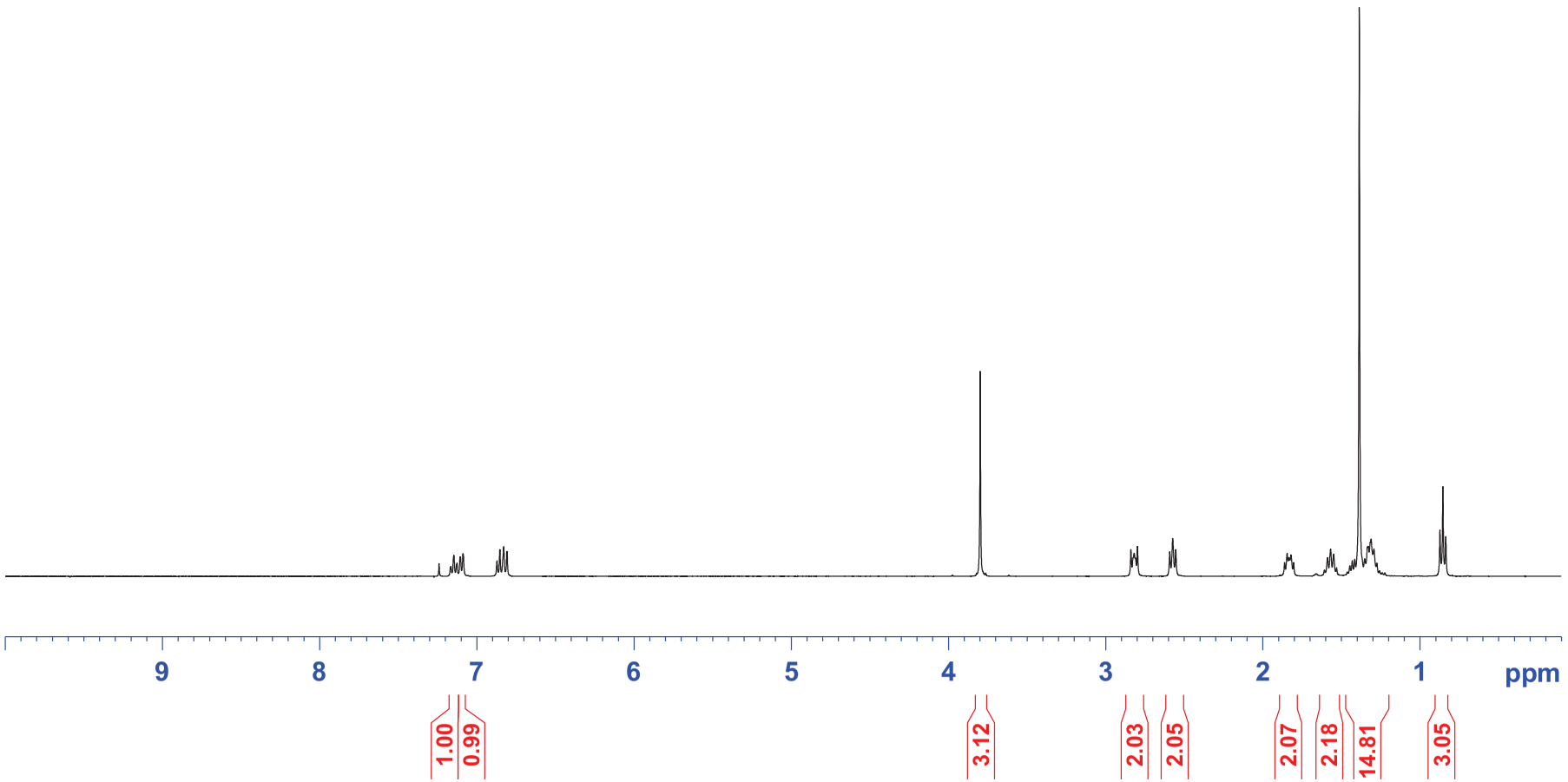


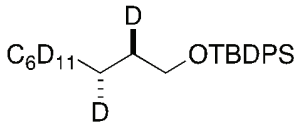
Table 3, entry 3



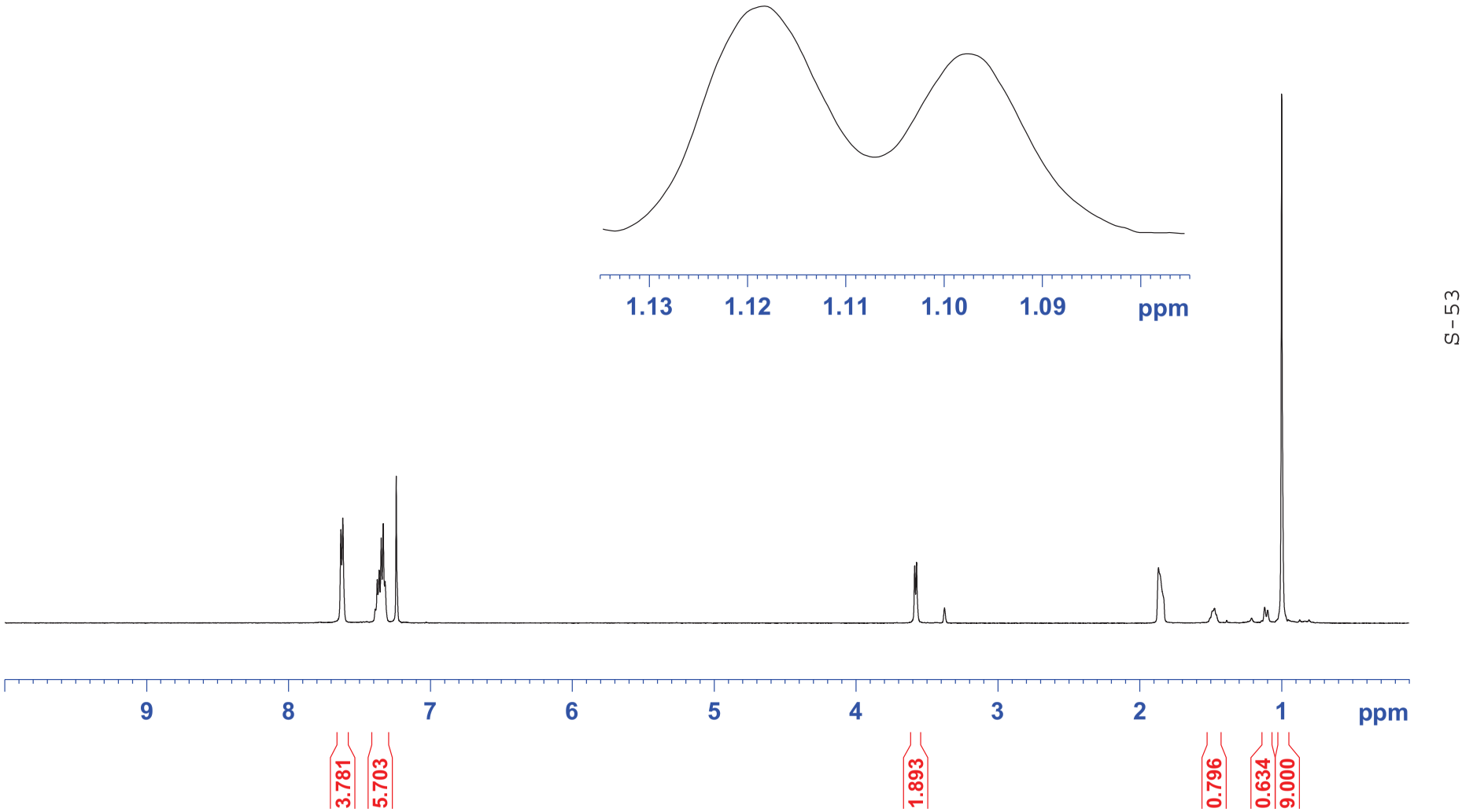

O
O
S
t-Bu
Et
OMe

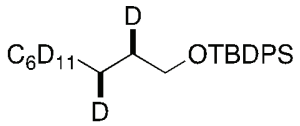
eq 11


9
8
7
6
5
4
3
2
1
ppm
1.00
0.99
3.12
2.03
2.05
2.07
2.18
14.81
3.05

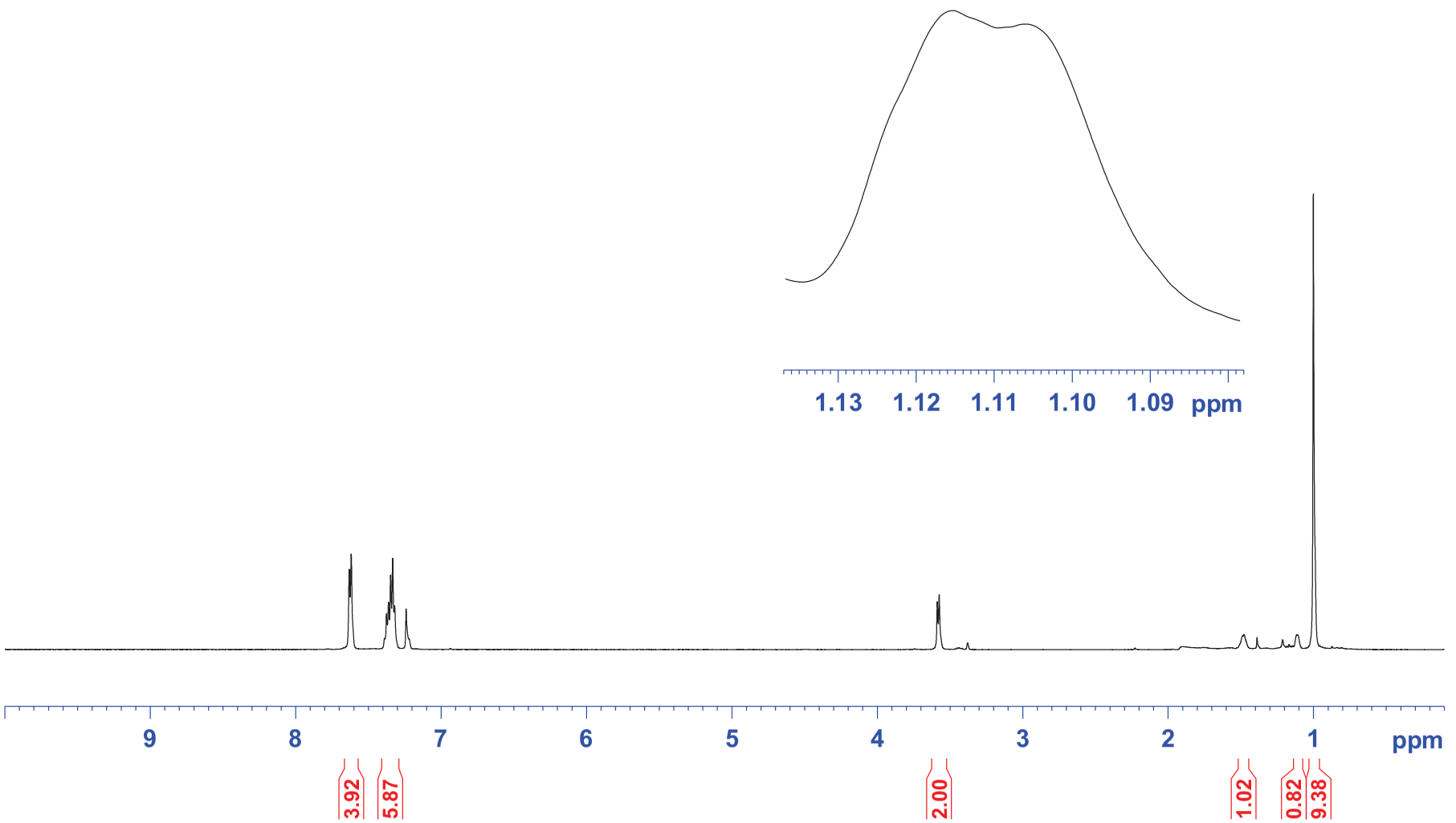

D
C6D11
OTBDPS
D

eq 12


1.13
1.12
1.11
1.10
1.09
ppm
9
8
7
6
5
4
3
2
1
ppm
3.781
5.703
1.893
0.796
0.634
9.000

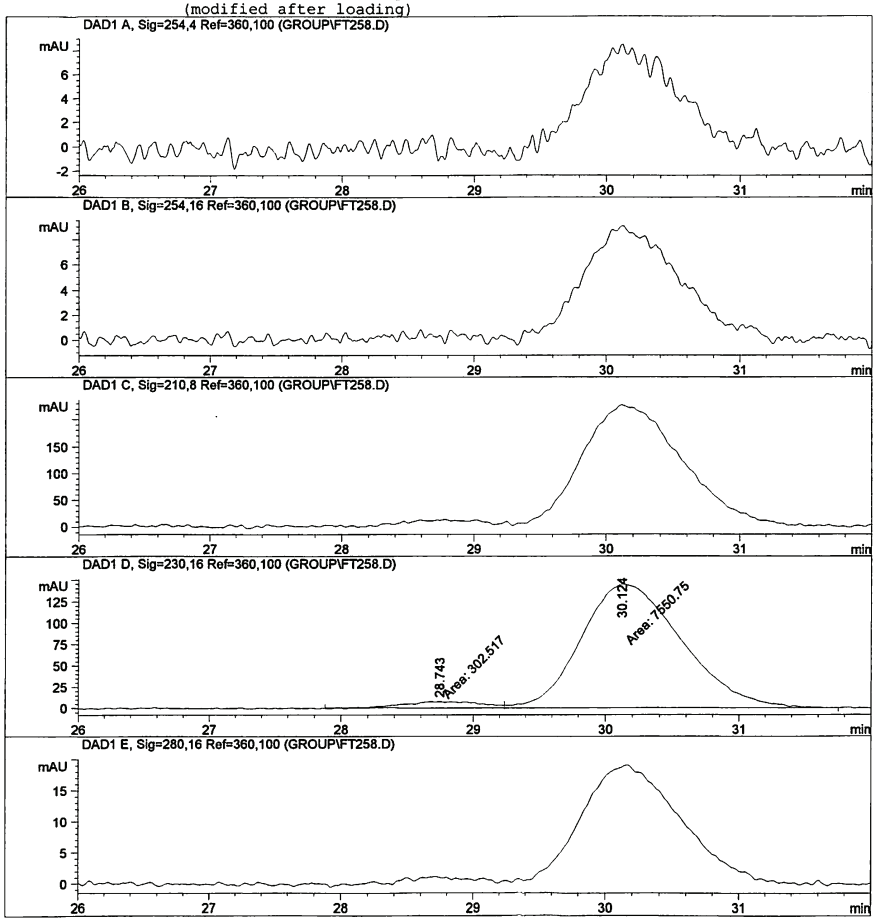

D
C6D11
OTBDPS
D

eq 13


1.13
1.12
1.11
1.10
1.09
ppm
9
8
7
6
5
4
3
2
1
ppm
3.92
5.87
2.00
1.02
0.82
9.38

Data File C:\HPCHEM\1\DATA\GROUP\FT258.D

```
==========================================================================
Injection Date  : 9/27/2010 4:01:21 PM          Seq. Line :   2
Sample Name     :                               Location : Vial 83
Acq. Operator   : JTM                             Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0540.M
Last changed    : 5/8/2009 8:38:12 AM by NM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0130.M
Last changed    : 3/15/2012 8:01:16 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT258.D

```
==========================================================
                      Area Percent Report
==========================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100


Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  28.743 MF    0.6693  302.51660    7.53312   3.8521
   2  30.124 FM    0.8707 7550.74756  144.52991  96.1479

Totals :                  7853.26416  152.06302

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                        *** End of Report ***
```

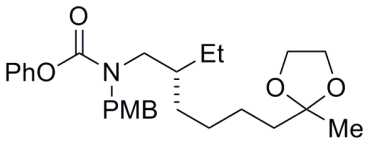


Table 1, entry 1

with (*R*,*R*)-DMPEDA

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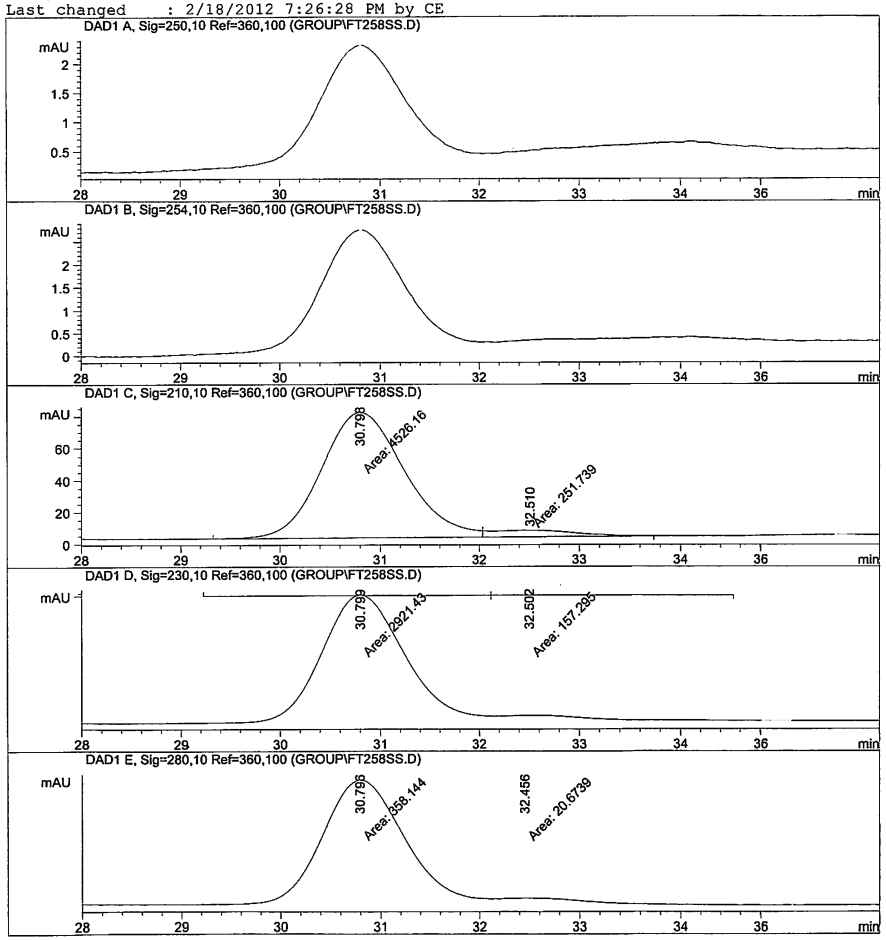
Data File C:\HPCHEM\1\DATA\GROUP\FT258SS.D

```
Injection Date  : 9/30/2010 6:19:36 PM          Seq. Line :  68
Sample Name     :                               Location : Vial 57
Acq. Operator   : JTM                             Inj :  1
Acq. Instrument : Instrument 1                Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-05-40.M
Last changed    : 8/18/2010 8:30:02 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\HDOD1.M
Last changed    : 2/18/2012 7:26:28 PM by CE
```



Data File C:\HPCHEM\1\DATA\GROUP\FT258SS.D

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100


Signal 2: DAD1 B, Sig=254,10 Ref=360,100


Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  30.798 MF    0.9596 4526.16113   78.61259  94.7312
   2  32.510 FM    0.9959  251.73897    4.21280   5.2688

Totals :                  4777.90010   82.82539

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  30.799 MF    0.9628 2921.43140   50.57107  94.8909
   2  32.502 FM    1.0303  157.29535    2.54455   5.1091

Totals :                  3078.72675   53.11563

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  30.796 MF    0.9523  358.14407    6.26779  94.5425
   2  32.456 FM    1.0343   20.67391 3.33154e-1   5.4575

Totals :                   378.81799    6.60095

 Results obtained with enhanced integrator!
```

*** End of Report ***

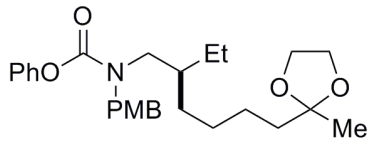
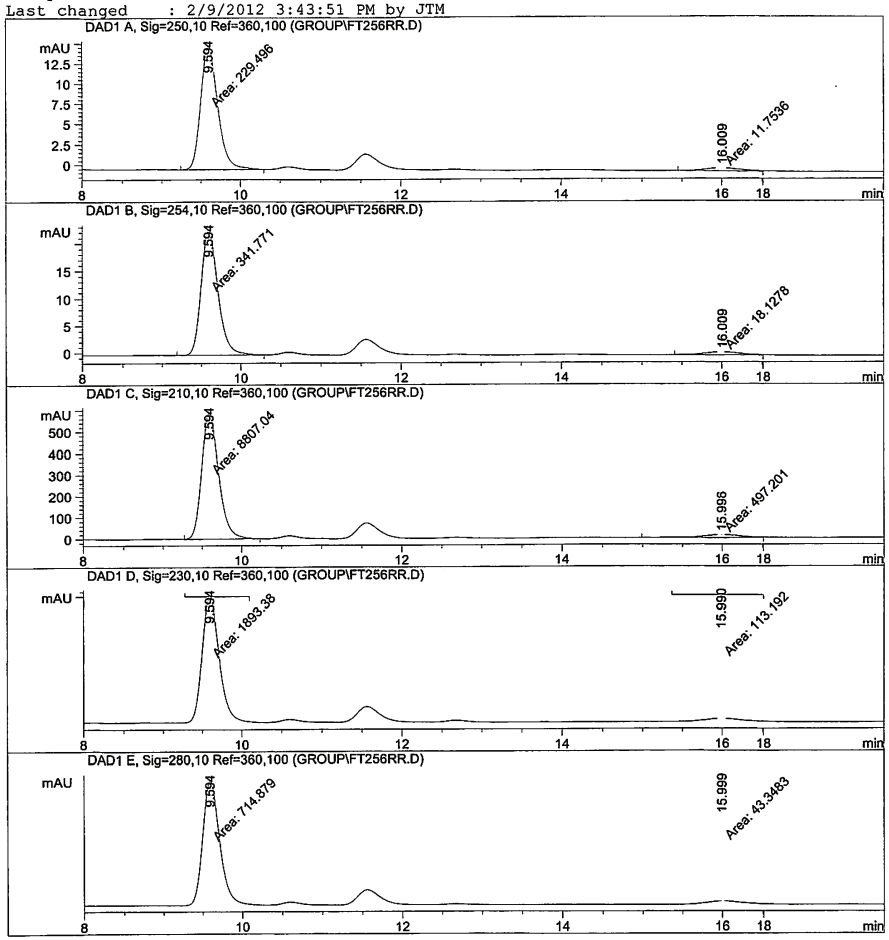


Table 1, entry 1

with (*S*,*S*)-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\FT256RR.D

```
==================================================================
Injection Date  : 9/30/2010 4:06:45 PM          Seq. Line :  62
Sample Name     :                               Location : Vial 52
Acq. Operator   : JTM                              Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-20.M
Last changed    : 8/20/2010 7:18:12 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 2/9/2012 3:43:51 PM by JTM
```



Data File C:\HPCHEM\1\DATA\GROUP\FT256RR.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.594 MM    0.2509  229.49628   15.24338  95.1280
   2  16.009 MM    0.5322   11.75363 3.68066e-1   4.8720

Totals :                   241.24991   15.61144

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.594 MM    0.2496  341.77097   22.81993  94.9631
   2  16.009 MM    0.5284   18.12779 5.71758e-1   5.0369

Totals :                   359.89876   23.39169

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.594 MM    0.2500 8807.04492  587.09119  94.6562
   2  15.996 MM    0.5840  497.20142   14.18899   5.3438

Totals :                  9304.24634  601.28018

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.594 MM    0.2488 1893.37683  126.84153  94.3589
   2  15.990 MM    0.5575  113.19214    3.38415   5.6411

Totals :                  2006.56897  130.22568

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.594 MM    0.2495  714.87854   47.76329  94.2829
   2  15.999 MM    0.5552   43.34834    1.30139   5.7171

Totals :                   758.22688   49.06468
```

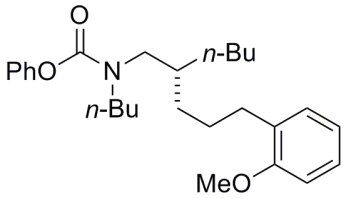
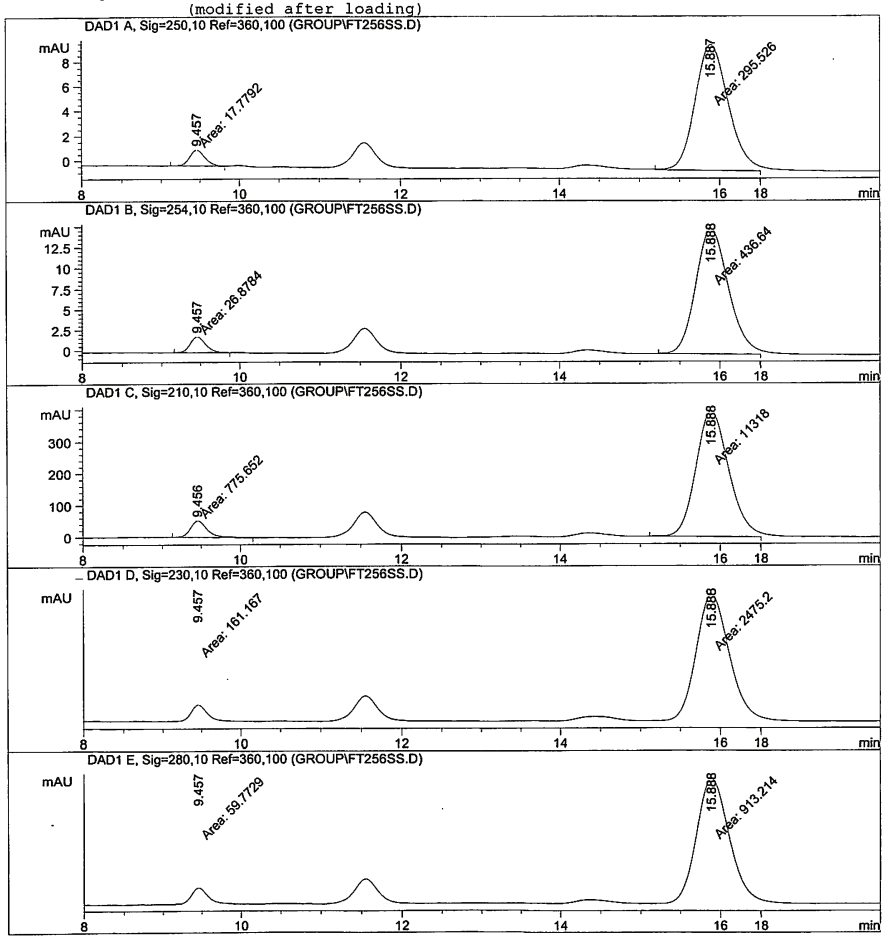


Table 1, entry 2

with (*R*,*R*)-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\FT256SS.D

```
Injection Date  : 9/30/2010 3:45:33 PM          Seq. Line :  61
Sample Name     :                               Location : Vial 51
Acq. Operator   : JTM                           Inj :  1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-20.M
Last changed    : 8/20/2010 7:18:12 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 9:12:08 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT256SS.D

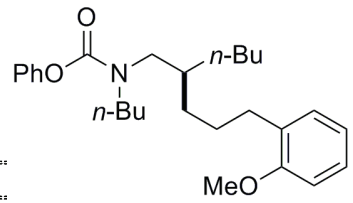


Table 1, entry 2

with (*S*,*S*)-DMPEDA

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.457	MM	0.2300	17.77925	1.28829	5.6747
2	15.887	MM	0.4829	295.52594	10.19978	94.3253
Totals :				313.30519	11.48807	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.457	MM	0.2318	26.87836	1.93254	5.7988
2	15.888	MM	0.4790	436.63989	15.19328	94.2012
Totals :				463.51825	17.12582	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.456	MM	0.2504	775.65216	51.62696	6.4137
2	15.888	MM	0.4798	1.13180e4	393.17139	93.5863
Totals :				1.20937e4	444.79835	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.457	MM	0.2429	161.16673	11.05778	6.1132
2	15.888	MM	0.4847	2475.20435	85.11755	93.8868
Totals :				2636.37108	96.17534	

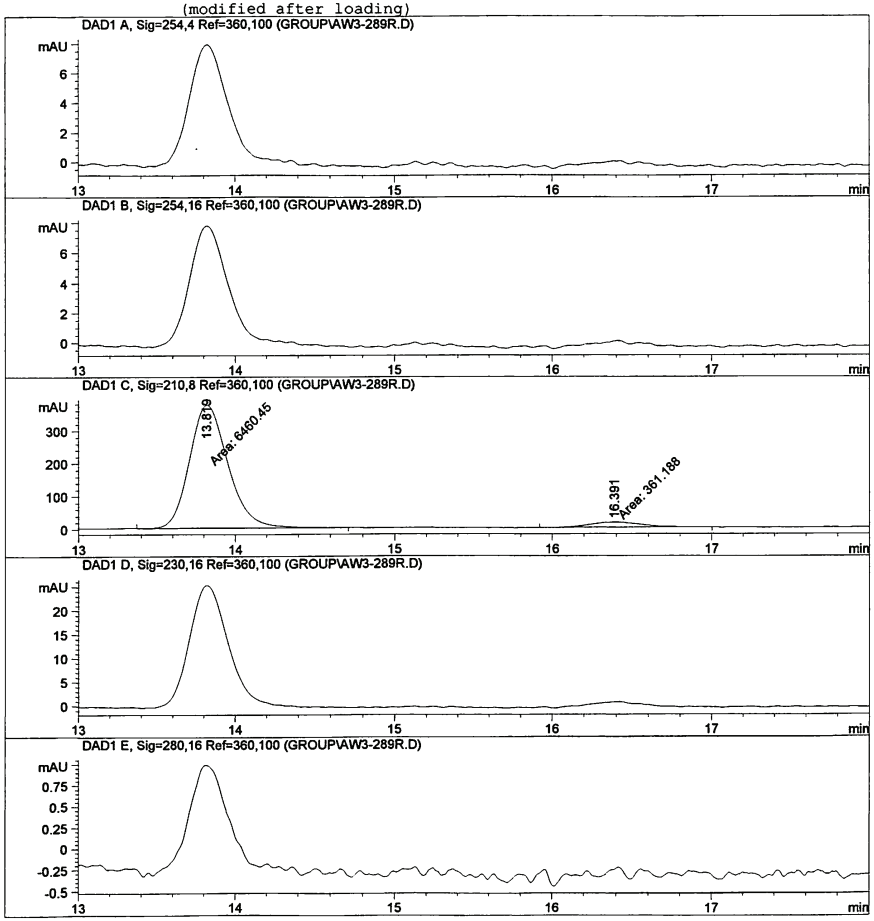
Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.457	MM	0.2442	59.77291	4.07927	6.1432
2	15.888	MM	0.4793	913.21375	31.75748	93.8568
Totals :				972.98665	35.83674	

Data File C:\HPCHEM\1\DATA\GROUP\AW3-289R.D

```
==========================================================================
Injection Date  : 5/30/2011 9:12:25 AM          Seq. Line :   4
Sample Name     :                               Location : Vial 13
Acq. Operator   : JTM                              Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0130.M
Last changed    : 5/30/2011 9:14:55 AM by JTM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0130.M
Last changed    : 3/16/2012 7:40:36 AM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-289R.D

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.819 MM    0.2882 6460.44727  373.64856  94.7053
   2  16.391 MM    0.3767  361.18805   15.97998   5.2947

Totals :                  6821.63531  389.62854

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100


Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                        *** End of Report ***
```

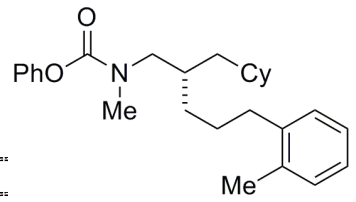
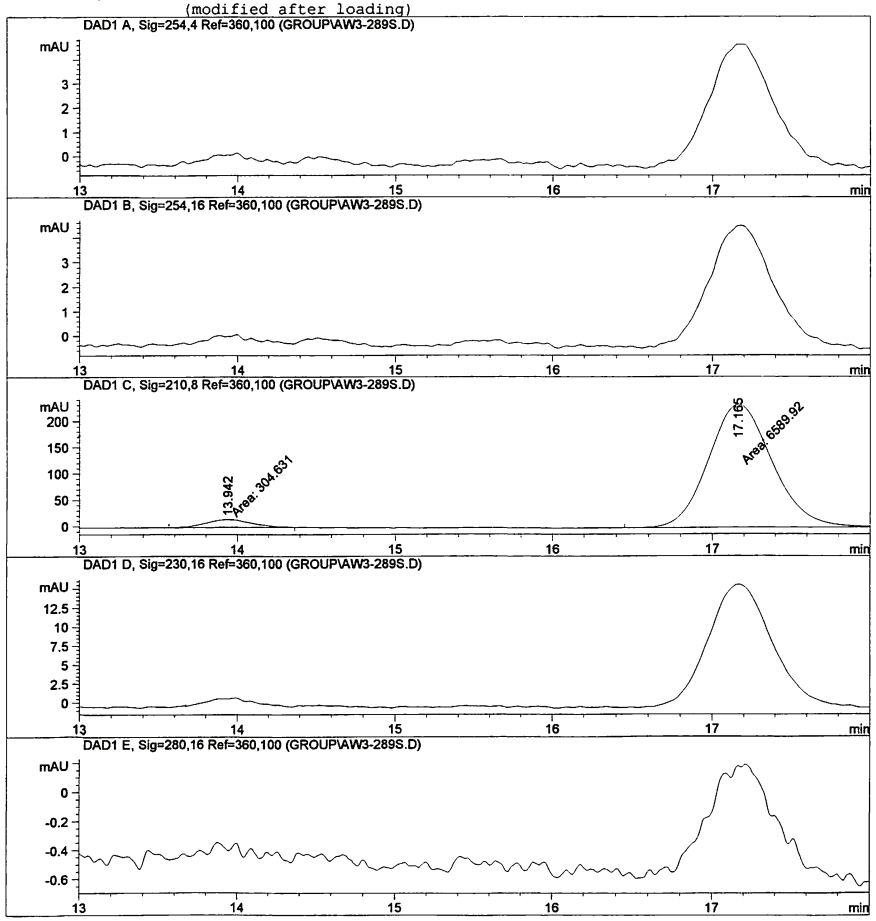


Table 1, entry 3

with (*R*,*R*)-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW3-289S.D

```
==========================================================================
Injection Date  : 5/30/2011 8:43:11 AM          Seq. Line :   3
Sample Name     :                               Location : Vial 12
Acq. Operator   : JTM                              Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0130.M
Last changed    : 5/30/2011 9:11:06 AM by JTM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0130.M
Last changed    : 3/16/2012 7:44:55 AM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-289S.D

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.942 MM    0.3422  304.63107   14.83871   4.4184
   2  17.165 MM    0.4701 6589.92285  233.61250  95.5816

Totals :                  6894.55392  248.45121

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100


Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                        *** End of Report ***
```

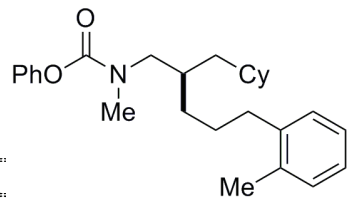


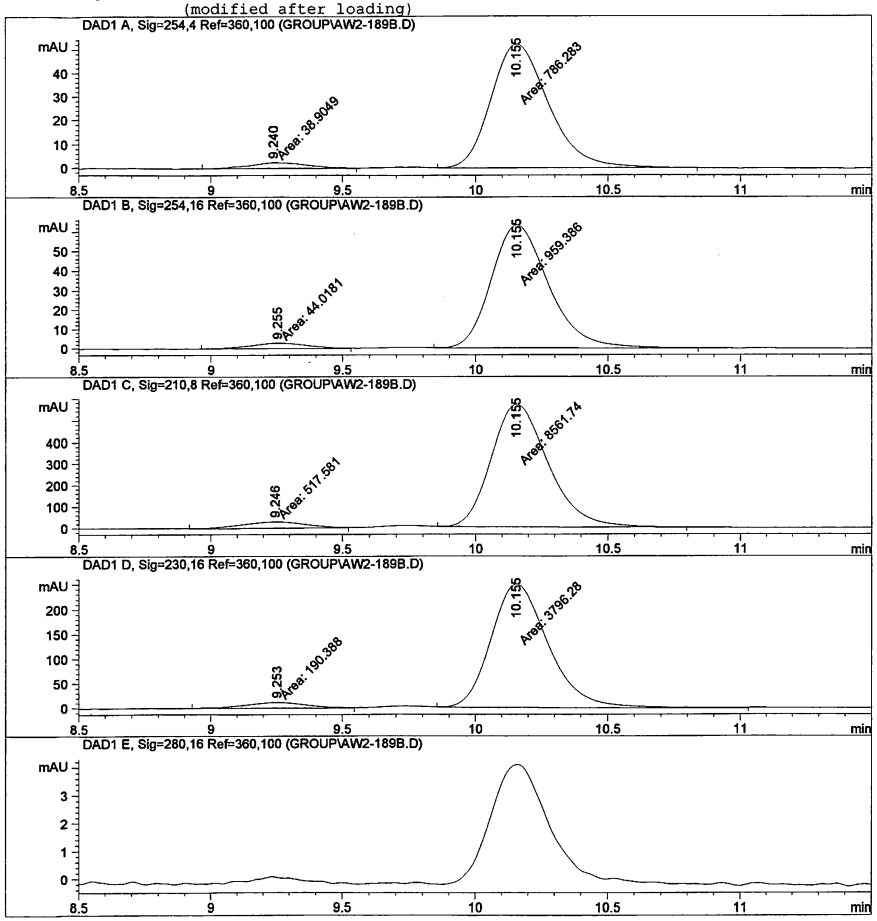
Table 1, entry 3

with (*S*,*S*)-DMPEDA

S-60

Data File C:\HPCHEM\1\DATA\GROUP\AW2-189B.D

```
==========================================================================
Injection Date  : 10/21/2010 6:33:27 PM          Seq. Line :   4
Sample Name     :                                 Location : Vial 7
Acq. Operator   : JTM                                Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0130.M
Last changed    : 5/8/2009 8:39:29 AM by NM
Analysis Method : C:\HPCHEM\1\METHODS\ADH-3060.M
Last changed    : 3/15/2012 2:34:48 PM by JTM
                  (modified after loading)
```



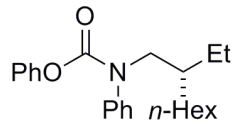


Table 1, entry 4

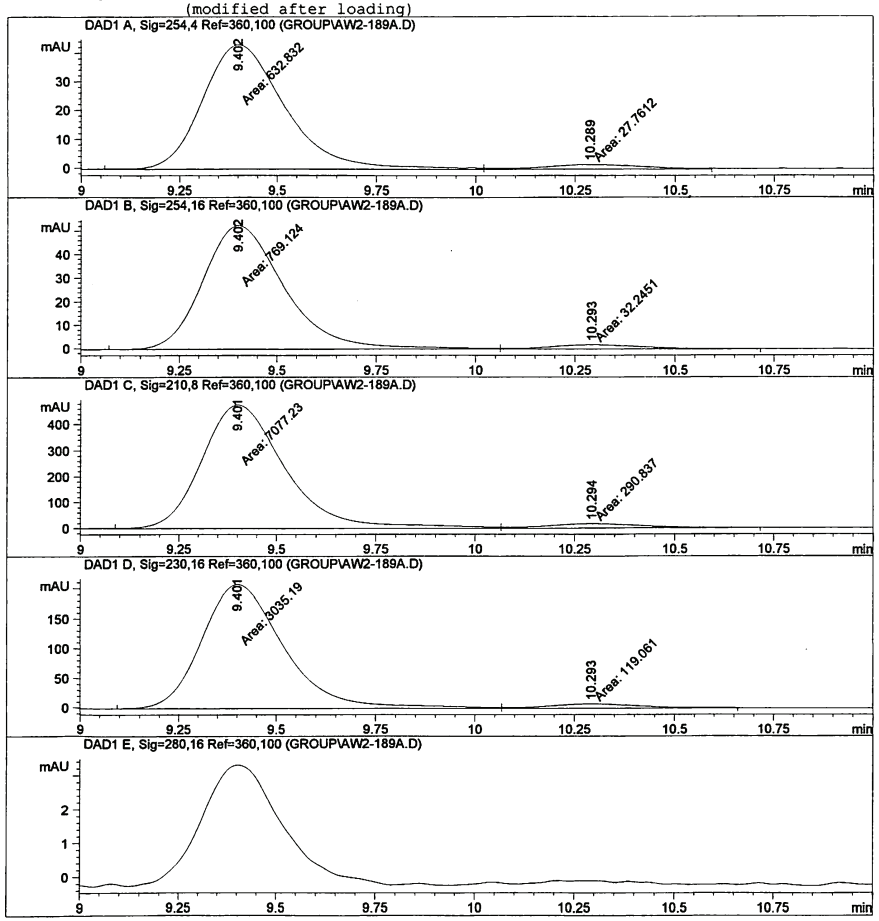
with (*R*,*R*)-DMPEDA

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.240 MM    0.2648   38.90487    2.44858   4.7147
   2  10.155 MM    0.2519  786.28302   52.02510  95.2853

Totals :                   825.18789   54.47368

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.255 MM    0.2573   44.01814    2.85161   4.3869
   2  10.155 MM    0.2522  959.38580   63.40101  95.6131

Totals :                  1003.40395   66.25262

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.246 MM    0.2895  517.58130   29.79925   5.7007
   2  10.155 MM    0.2517 8561.73828  566.92328  94.2993

Totals :                  9079.31958  596.72253

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.253 MM    0.2694  190.38773   11.77664   4.7756
   2  10.155 MM    0.2536 3796.27905  249.46820  95.2244

Totals :                  3986.66678  261.24484

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                        *** End of Report ***
```

S-61

Data File C:\HPCHEM\1\DATA\GROUP\AW2-189A.D

```
==========================================================================
Injection Date  : 10/21/2010 6:02:11 PM          Seq. Line :   3
Sample Name     :                                 Location : Vial 6
Acq. Operator   : JTM                                Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0130.M
Last changed    : 5/8/2009 8:39:29 AM by NM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0130.M
Last changed    : 3/16/2012 7:44:55 AM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW2-189A.D

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.402 MF    0.2437  632.83179   43.28032  95.7975
   2  10.289 FM    0.2879   27.76118    1.60704   4.2025

Totals :                   660.59296   44.88737

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.402 MF    0.2432  769.12445   52.69825  95.9762
   2  10.293 FM    0.2792   32.24513    1.92468   4.0238

Totals :                   801.36958   54.62293

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.401 MF    0.2477 7077.23047  476.24072  96.0527
   2  10.294 FM    0.2780  290.83670   17.43646   3.9473

Totals :                  7368.06717  493.67719

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.401 MF    0.2440 3035.19312  207.28575  96.2254
   2  10.293 FM    0.2691  119.06054    7.37405   3.7746

Totals :                  3154.25365  214.65980

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                        *** End of Report ***
```

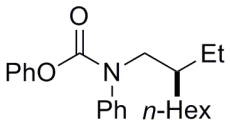
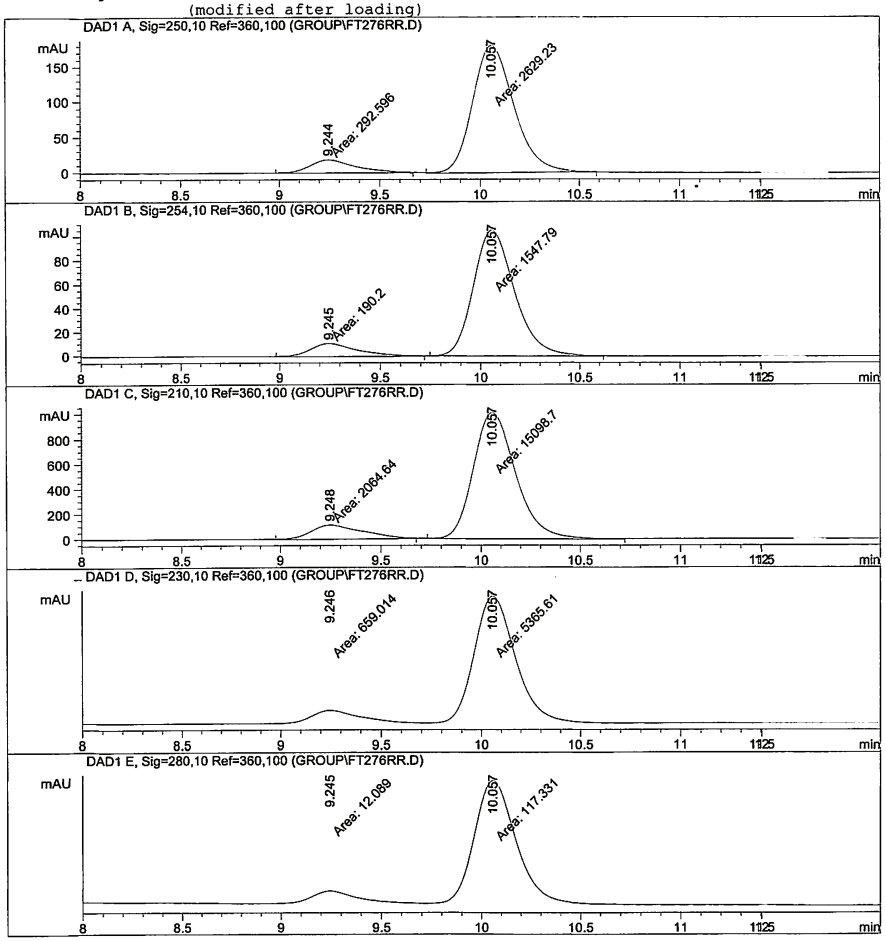


Table 1, entry 4

with (*S*,*S*)-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\FT276RR.D

```
==================================================================
Injection Date  : 9/29/2010 2:49:52 PM          Seq. Line :   9
Sample Name     :                               Location : Vial 43
Acq. Operator   : JTM                             Inj :   1
Acq. Instrument : Instrument 1                Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 7/31/2010 2:44:37 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\YL-AD10.M
Last changed    : 3/15/2012 3:20:45 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT276RR.D

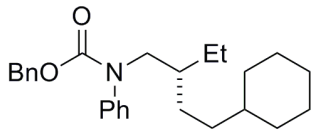


Table 1, entry 5

with (*R*,*R*)-DMPEDA

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.244 MM    0.2637  292.59586   18.49278  10.0142
   2  10.057 MM    0.2420 2629.22705  181.08366  89.9858

Totals :                  2921.82291  199.57645

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.245 MM    0.2826  190.19994   11.21728  10.9437
   2  10.057 MM    0.2432 1547.78943  106.05862  89.0563

Totals :                  1737.98936  117.27590

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.248 MM    0.3009 2064.63843  114.35263  12.0293
   2  10.057 MM    0.2497 1.50987e4  1007.75421  87.9707

Totals :                  1.71634e4  1122.10684

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.246 MM    0.2862  659.01404   38.37855  10.9387
   2  10.057 MM    0.2427 5365.61426  368.48212  89.0613

Totals :                  6024.62830  406.86067

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.245 MM    0.2494   12.08900 8.07831e-1   9.3409
   2  10.057 MM    0.2438  117.33055    8.01963  90.6591

Totals :                   129.41955    8.82746
```

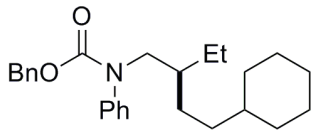
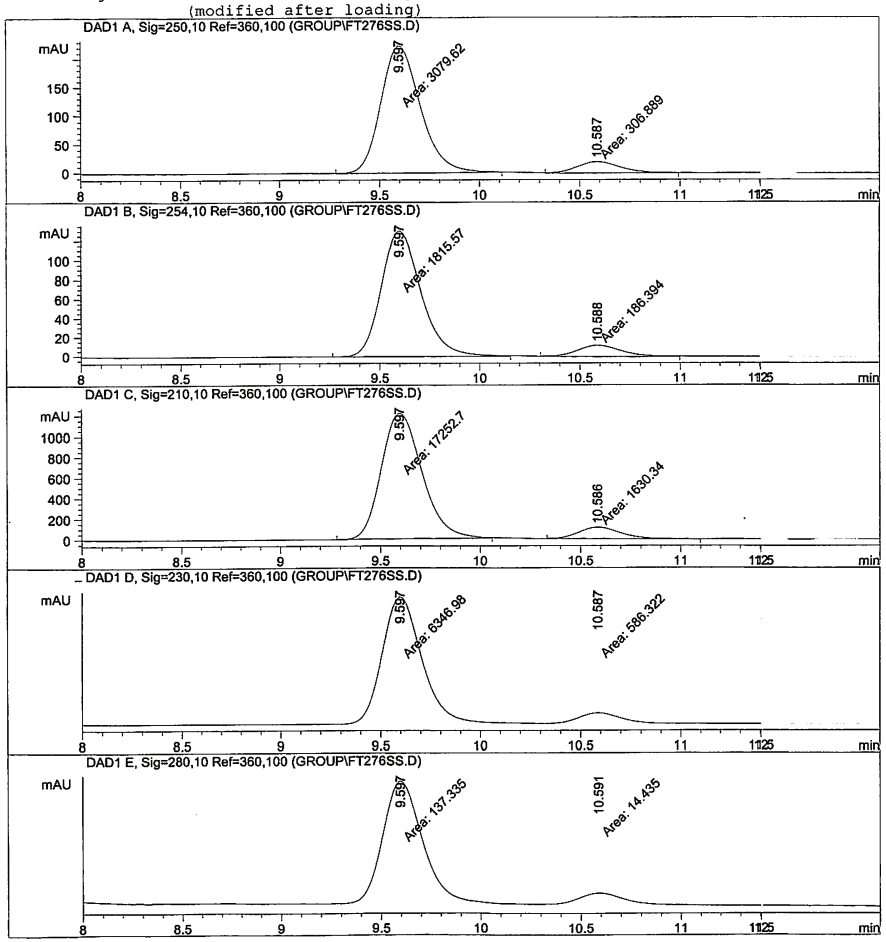


Table 1, entry 5

with (*S*,*S*)-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\FT276SS.D

```
Injection Date  : 9/29/2010 2:18:40 PM          Seq. Line :   8
Sample Name     :                               Location : Vial 42
Acq. Operator   : JTM                             Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 7/31/2010 2:44:37 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\YL-AD10.M
Last changed    : 3/15/2012 3:16:06 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT276SS.D

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.597	MM	0.2289	3079.61865	224.21060	90.9379
2	10.587	MM	0.2548	306.88901	20.07479	9.0621
Totals :				3386.50766	244.28539	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.597	MM	0.2303	1815.56946	131.40201	90.6894
2	10.588	MM	0.2602	186.39389	11.94097	9.3106
Totals :				2001.96335	143.34298	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.597	MM	0.2369	1.72527e4	1213.95508	91.3661
2	10.586	MM	0.2440	1630.34290	111.37382	8.6339
Totals :				1.88831e4	1325.32890	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.597	MM	0.2301	6346.98486	459.76541	91.5434
2	10.587	MM	0.2487	586.32239	39.28577	8.4566
Totals :				6933.30725	499.05119	

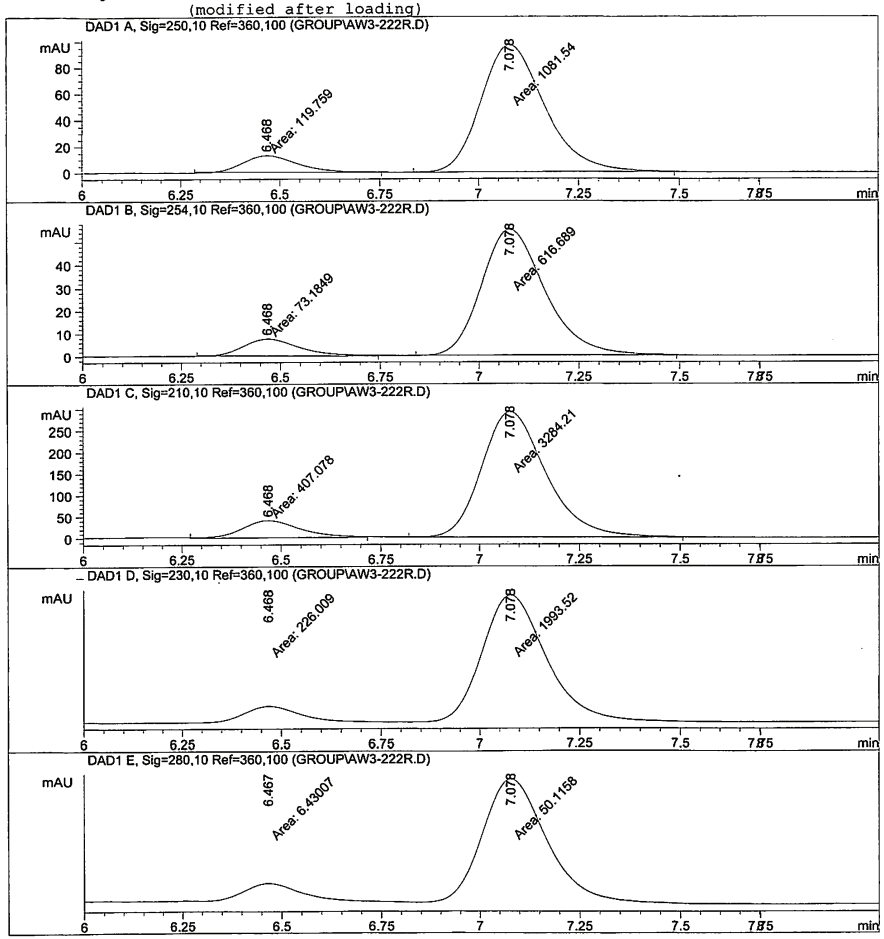
Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

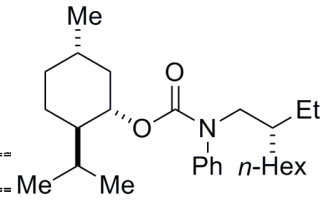
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.597	MM	0.2313	137.33536	9.89728	90.4889
2	10.591	MM	0.2538	14.43499	9.47960e-1	9.5111
Totals :				151.77035	10.84524	

Data File C:\HPCHEM\1\DATA\GROUP\AW3-222R.D

```
==========================================================================
Injection Date  : 4/24/2011 11:26:55 AM          Seq. Line :   5
Sample Name     :                                 Location : Vial 93
Acq. Operator   : NB                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 3/15/2011 7:02:33 AM by YL
Analysis Method : C:\HPCHEM\1\METHODS\YL-AD10.M
Last changed    : 3/15/2012 3:20:45 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-222R.D



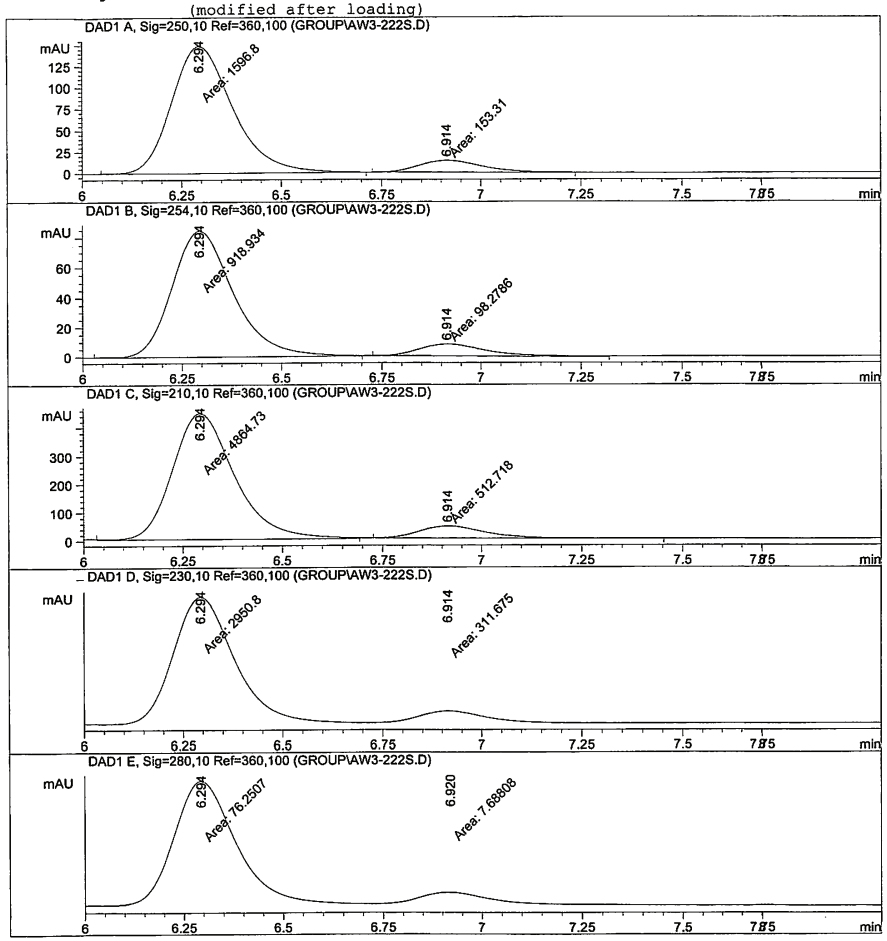
eq 2

with (*R*,*R*)-DMPEDA

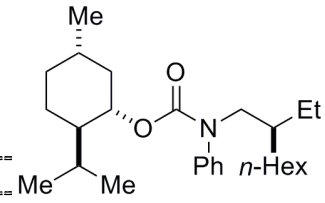
```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.468 MM    0.1583  119.75882   12.60619   9.9691
   2   7.078 MM    0.1860 1081.54041   96.93636  90.0309

Totals :                   1201.29922  109.54254

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.468 MM    0.1637   73.18491    7.45229  10.6084
   2   7.078 MM    0.1861  616.68933   55.23767  89.3916

Totals :                    689.87424   62.68996

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.468 MM    0.1696  407.07831   40.00994  11.0281
   2   7.078 MM    0.1876 3284.20996  291.69717  88.9719

Totals :                   3691.28827  331.70712

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.468 MM    0.1615  226.00859   23.32026  10.1827
   2   7.078 MM    0.1855 1993.51978  179.15785  89.8173

Totals :                   2219.52837  202.47812

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.467 MM    0.1619    6.43007 6.62005e-1  11.3714
   2   7.078 MM    0.1863   50.11582    4.48292  88.6286

Totals :                     56.54589    5.14493
```

Data File C:\HPCHEM\1\DATA\GROUP\AW3-222S.D

```
Injection Date  : 4/24/2011 10:55:43 AM          Seq. Line :   4
Sample Name     :                                 Location : Vial 92
Acq. Operator   : NB                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 3/15/2011 7:02:33 AM by YL
Analysis Method : C:\HPCHEM\1\METHODS\YL-AD10.M
Last changed    : 3/15/2012 3:31:56 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-222S.D



eq 3

with (*S*,*S*)-DMPEDA

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.294	MM	0.1788	1596.80164	148.83705	91.2400
2	6.914	MM	0.1849	153.31033	13.81947	8.7600
Totals :				1750.11197	162.65652	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.294	MM	0.1801	918.93365	85.02588	90.3384
2	6.914	MM	0.2013	98.27861	8.13677	9.6616
Totals :				1017.21227	93.16265	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.294	MM	0.1821	4864.72949	445.23761	90.4654
2	6.914	MM	0.1970	512.71790	43.37631	9.5346
Totals :				5377.44739	488.61392	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.294	MM	0.1783	2950.79907	275.83905	90.4467
2	6.914	MM	0.1945	311.67496	26.71047	9.5533
Totals :				3262.47403	302.54952	

Results obtained with enhanced integrator!

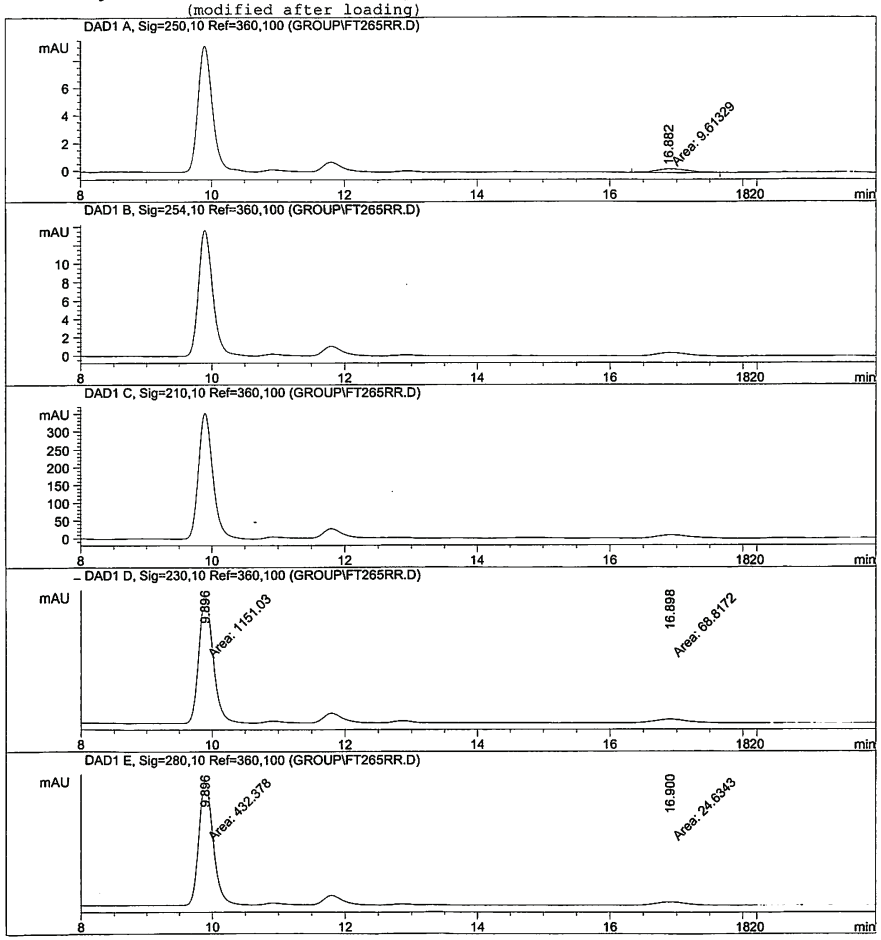
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.294	MM	0.1824	76.25069	6.96575	90.8409
2	6.920	MM	0.1965	7.68808	6.51966e-1	9.1591
Totals :				83.93876	7.61771	

Results obtained with enhanced integrator!

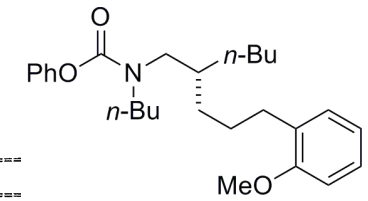
Data File C:\HPCHEM\1\DATA\GROUP\FT265RR.D

```
==================================================================
Injection Date  : 10/18/2010 10:48:03 PM          Seq. Line :   5
Sample Name     :                                  Location : Vial 41
Acq. Operator   : JTM                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 10/4/2010 10:50:43 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 9:36:17 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT265RR.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.882 MM    0.5756    9.61329 2.78345e-1 100.0000

Totals :                     9.61329 2.78345e-1

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100


Signal 3: DAD1 C, Sig=210,10 Ref=360,100


Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.896 MM    0.2518 1151.02942   76.18850  94.3585
   2  16.898 MM    0.5357   68.81721    2.14108   5.6415

Totals :                  1219.84663   78.32958

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.896 MM    0.2523  432.37845   28.56366  94.6097
   2  16.900 MM    0.5278   24.63426 7.77940e-1   5.3903

Totals :                   457.01271   29.34160

 Results obtained with enhanced integrator!
==================================================================
                      *** End of Report ***
```

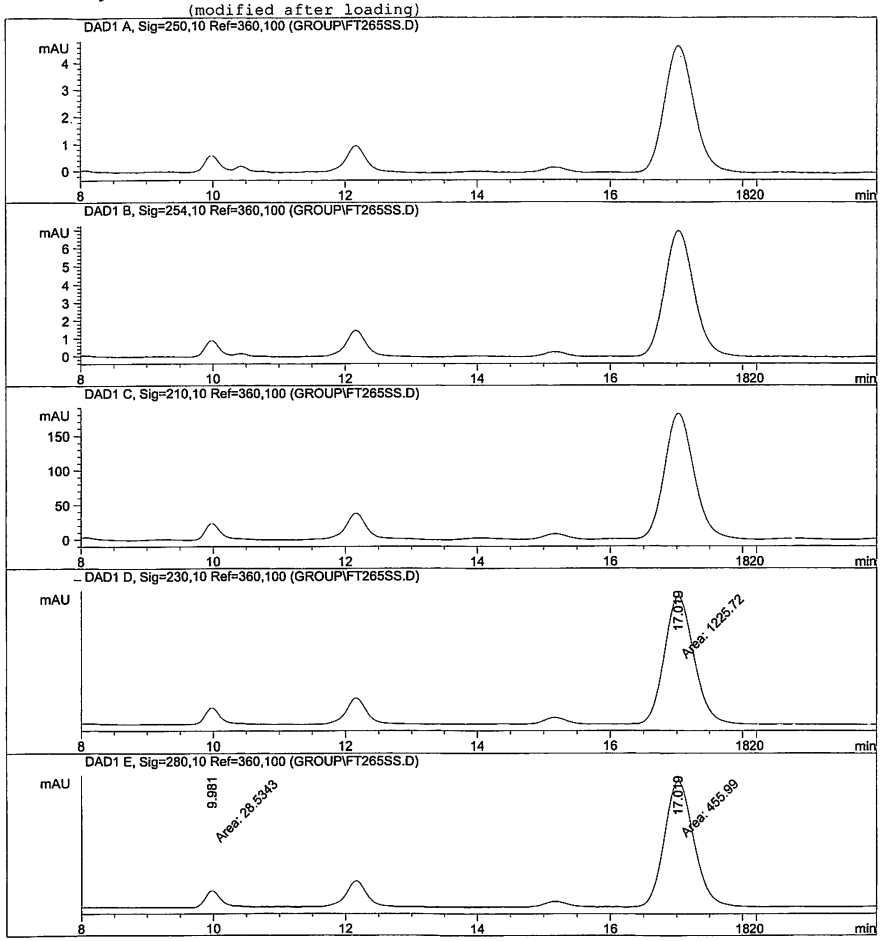


eq 4

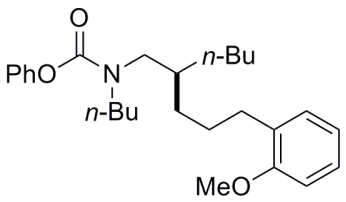
with (*R*,*R*)-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\FT265SS.D

```
==================================================================
Injection Date  : 10/18/2010 11:19:12 PM         Seq. Line :   6
Sample Name     :                                 Location : Vial 31
Acq. Operator   : JTM                                Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 10/4/2010 10:50:43 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 9:41:19 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT265SS.D



eq 4

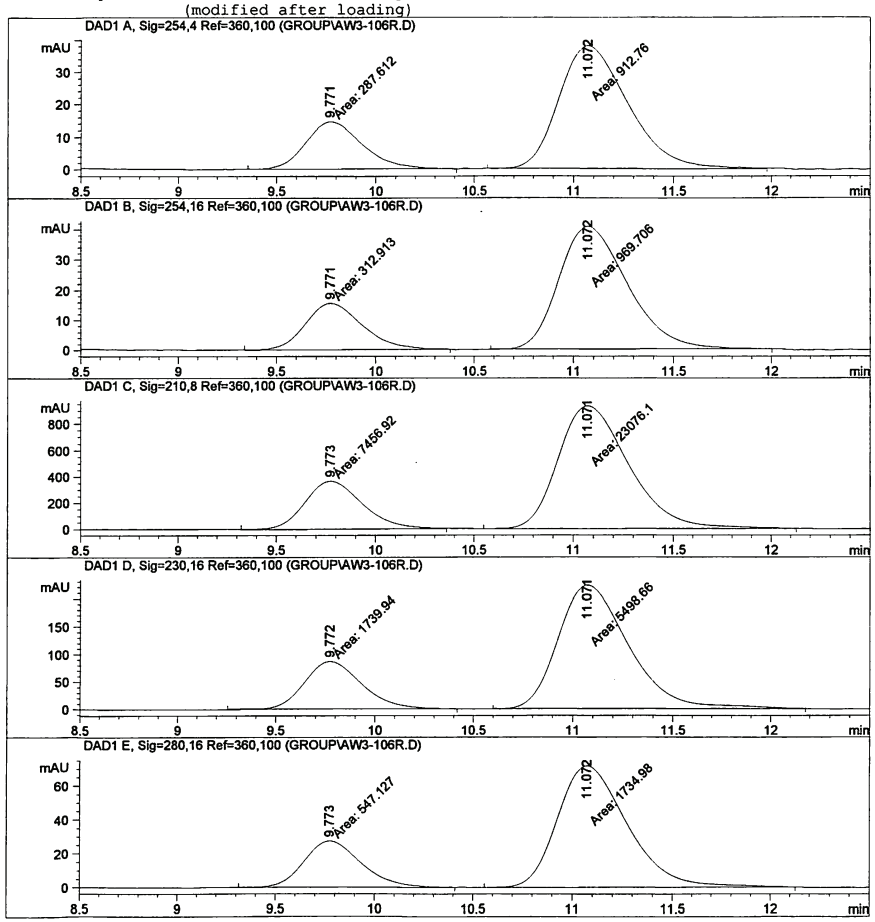
with (*S*,*S*)-DMPEDA

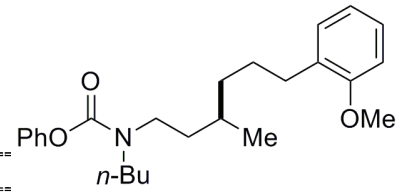
```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100


Signal 2: DAD1 B, Sig=254,10 Ref=360,100


Signal 3: DAD1 C, Sig=210,10 Ref=360,100


Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.019 MM    0.5195 1225.71704   39.32386 100.0000

Totals :                  1225.71704   39.32386

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.981 MM    0.2553   28.53433    1.86265   5.8891
   2  17.019 MM    0.5197  455.99030   14.62324  94.1109

Totals :                   484.52462   16.48589

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

S-89

Data File C:\HPCHEM\1\DATA\GROUP\AW3-106R.D

```
==========================================================================
Injection Date  : 2/25/2011 8:39:51 PM          Seq. Line :  11
Sample Name     :                               Location : Vial 83
Acq. Operator   : JTM                              Inj :  1
Acq. Instrument : Instrument 1                 Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ODH-0530.M
Last changed    : 2/24/2011 1:43:37 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\ADH-3060.M
Last changed    : 3/15/2012 2:44:26 PM by JTM
                  (modified after loading)
```





eq 5

with (*R*,*R*)-DMPEDA

```
==========================================================
                    Area Percent Report
==========================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.771	MM	0.3319	287.61221	14.44424	23.9602
2	11.072	MM	0.4006	912.76019	37.97793	76.0398
Totals :				1200.37241	52.42217	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.771	MM	0.3340	312.91330	15.61490	24.3964
2	11.072	MM	0.3971	969.70630	40.70020	75.6036
Totals :				1282.61960	56.31509	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.773	MM	0.3386	7456.91504	367.03772	24.4225
2	11.071	MM	0.4110	2.30761e4	935.83020	75.5775
Totals :				3.05330e4	1302.86792	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.772	MM	0.3356	1739.93542	86.41921	24.0369
2	11.071	MM	0.4118	5498.66113	222.55084	75.9631
Totals :				7238.59656	308.97005	

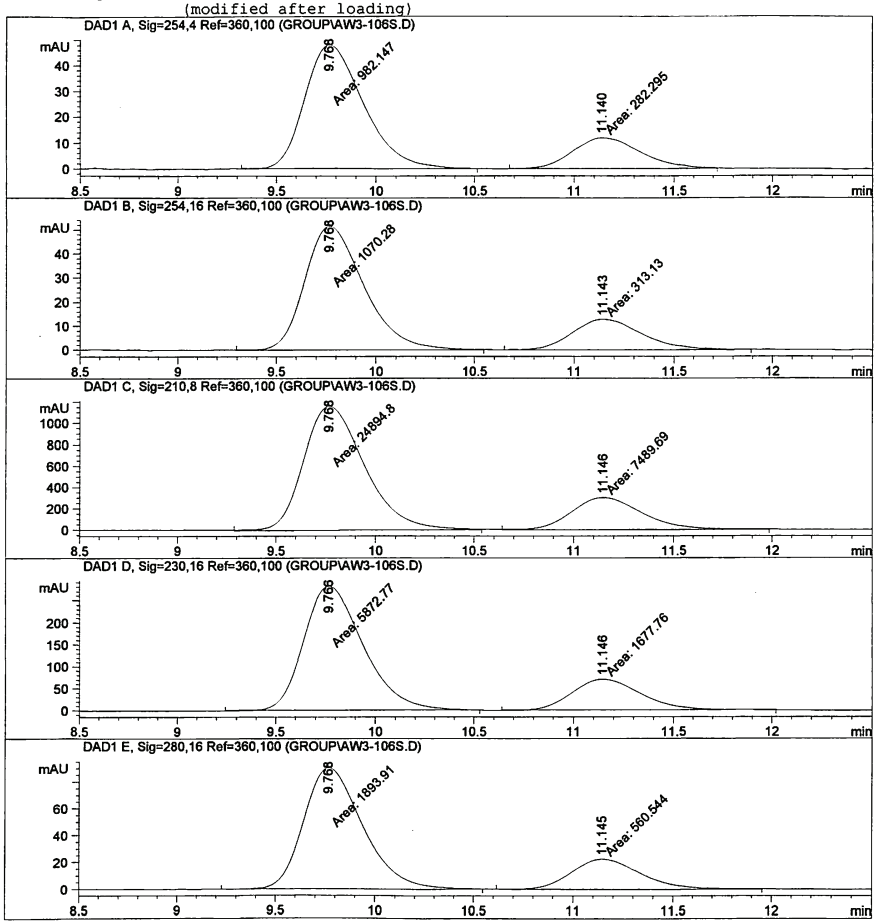
Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

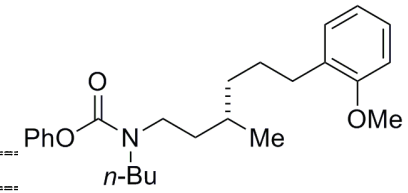
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.773	MM	0.3335	547.12738	27.33938	23.9746
2	11.072	MM	0.4034	1734.98340	71.67774	76.0254
Totals :				2282.11078	99.01712	

Data File C:\HPCHEM\1\DATA\GROUP\AW3-106S.D

```
==========================================================================
Injection Date  : 2/25/2011 8:08:37 PM          Seq. Line :  10
Sample Name     :                               Location : Vial 82
Acq. Operator   : JTM                              Inj :  1
Acq. Instrument : Instrument 1                 Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ODH-0530.M
Last changed    : 2/24/2011 1:43:37 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\ADH-3060.M
Last changed    : 3/15/2012 2:44:26 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-106S.D



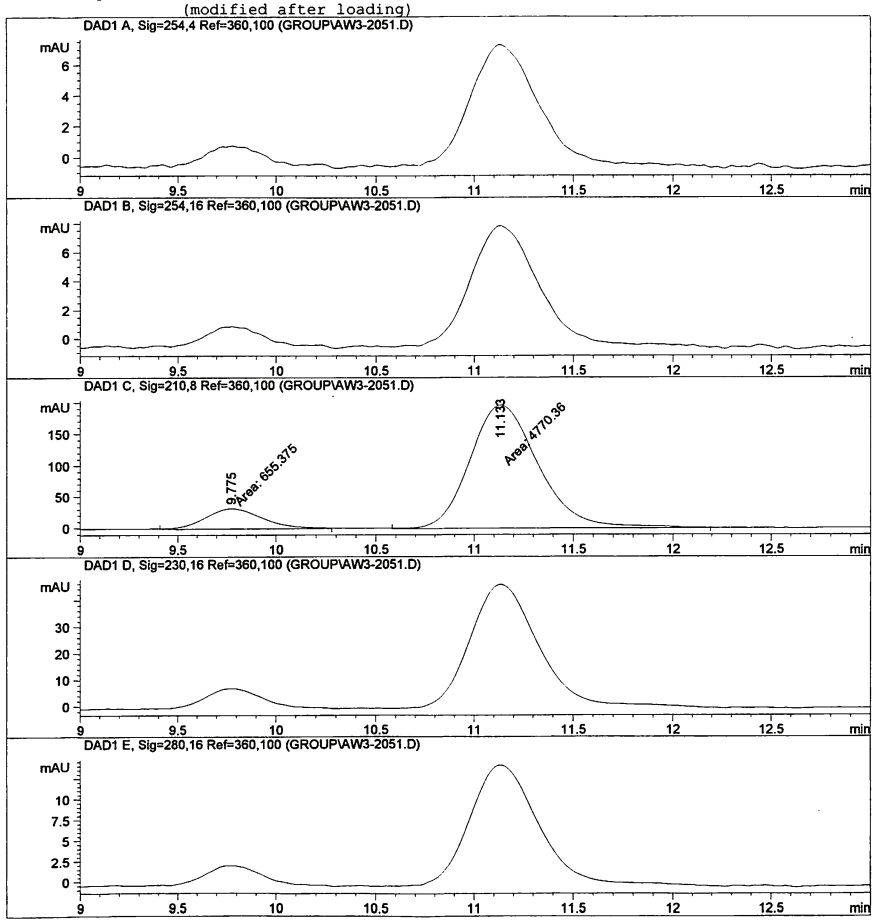
eq 5

with (*S*,*S*)-DMPEDA

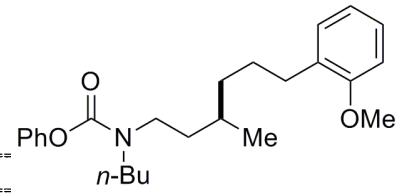
```
============================================================
                    Area Percent Report
============================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.768 MM    0.3414  982.14661   47.95120  77.6743
   2  11.140 MM    0.3955  282.29501   11.89659  22.3257

Totals :                  1264.44162   59.84779

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.768 MM    0.3449 1070.28064   51.72630  77.3654
   2  11.143 MM    0.4052  313.13013   12.87910  22.6346

Totals :                  1383.41077   64.60540

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.768 MM    0.3566 2.48948e4  1163.51208  76.8726
   2  11.146 MM    0.4113 7489.68652  303.50439  23.1274

Totals :                  3.23845e4  1467.01648

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.766 MM    0.3478 5872.77246  281.38940  77.7795
   2  11.146 MM    0.3968 1677.76440   70.47554  22.2205

Totals :                  7550.53687  351.86494

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.768 MM    0.3458 1893.91235   91.28130  77.1622
   2  11.145 MM    0.4119  560.54370   22.68028  22.8378

Totals :                  2454.45605  113.96158
```

Data File C:\HPCHEM\1\DATA\GROUP\AW3-2051.D

```
==========================================================================
Injection Date  : 4/14/2011 1:52:13 PM          Seq. Line :   2
Sample Name     :                               Location : Vial 61
Acq. Operator   : JTM                              Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ODH-0530.M
Last changed    : 2/24/2011 1:43:37 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0130.M
Last changed    : 3/16/2012 7:51:23 AM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-2051.D



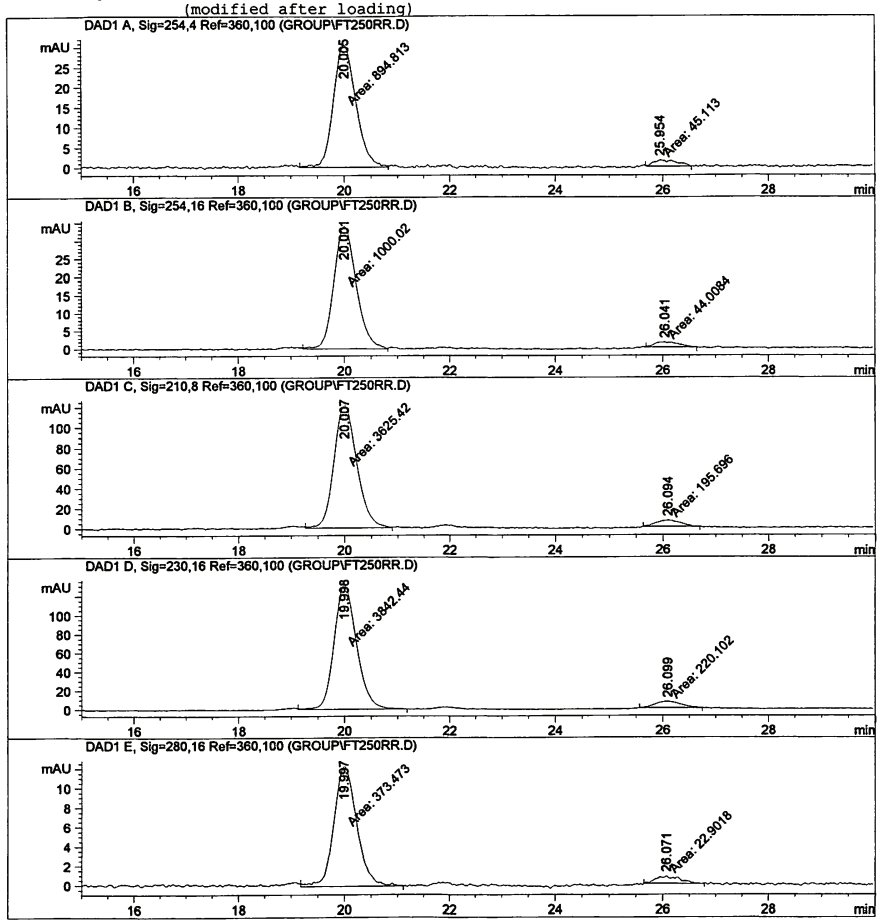
eq 5

with (*R*,*R*)-*m*-CF_3-DMPEDA

```
==========================================================
                    Area Percent Report
==========================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.775 MM    0.3381  655.37500   32.30318  12.0790
   2  11.133 MM    0.4043 4770.35791  196.67076  87.9210

Totals :                  5425.73291  228.97395

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100


Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                        *** End of Report ***
```

Data File C:\HPCHEM\1\DATA\GROUP\FT250RR.D

```
Injection Date  : 9/9/2010 1:19:00 AM          Seq. Line :   9
Sample Name     :                              Location : Vial 74
Acq. Operator   : jtm                             Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0530.M
Last changed    : 5/8/2009 8:37:41 AM by NM
Analysis Method : C:\HPCHEM\1\METHODS\NVOJ40A.M
Last changed    : 9/9/2010 9:39:59 AM by jtm
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT250RR.D

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.005	MM	0.4863	894.81317	30.66657	95.2004
2	25.954	MM	0.4732	45.11304	1.58910	4.7996
Totals :				939.92621	32.25567	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.001	MM	0.4867	1000.02008	34.24819	95.7848
2	26.041	MM	0.5098	44.00837	1.43881	4.2152
Totals :				1044.02845	35.68700	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.007	MM	0.5000	3625.41943	120.83723	94.8786
2	26.094	MM	0.5218	195.69553	6.25111	5.1214
Totals :				3821.11496	127.08834	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.998	MM	0.4915	3842.44141	130.30785	94.5822
2	26.099	MM	0.5503	220.10202	6.66655	5.4178
Totals :				4062.54343	136.97440	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.997	MM	0.5024	373.47284	12.38881	94.2222
2	26.071	MM	0.4987	22.90176	7.65352e-1	5.7778
Totals :				396.37460	13.15417	

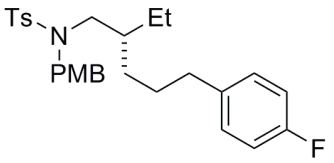
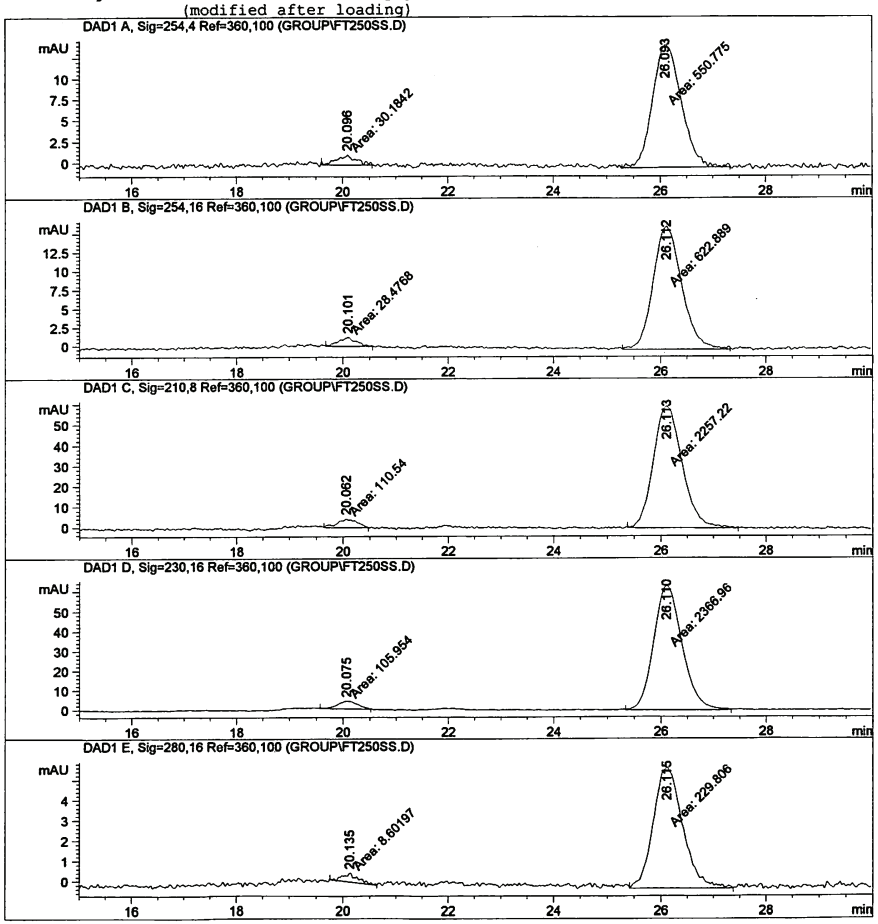


Table 2, entry 1

with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\FT250SS.D

```
==================================================================
Injection Date  : 9/9/2010 12:47:38 AM          Seq. Line :   8
Sample Name     :                               Location : Vial 73
Acq. Operator   : jtm                              Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0530.M
Last changed    : 5/8/2009 8:37:41 AM by NM
Analysis Method : C:\HPCHEM\1\METHODS\NVOJ40A.M
Last changed    : 9/9/2010 9:17:38 AM by jtm
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT250SS.D

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.096	MM	0.4177	30.18417	1.20445	5.1956
2	26.093	MM	0.6281	550.77478	14.61436	94.8044
Totals :				580.95895	15.81880	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.101	MM	0.3791	28.47677	1.25201	4.3719
2	26.112	MM	0.6299	622.88904	16.48125	95.6281
Totals :				651.36580	17.73326	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.062	MM	0.4288	110.53996	4.29608	4.6685
2	26.113	MM	0.6327	2257.22217	59.45892	95.3315
Totals :				2367.76212	63.75500	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.075	MM	0.4443	105.95383	3.97465	4.2846
2	26.110	MM	0.6269	2366.96021	62.92990	95.7154
Totals :				2472.91404	66.90455	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.135	MM	0.3284	8.60197	4.36596e-1	3.6081
2	26.115	MM	0.6409	229.80605	5.97579	96.3919
Totals :				238.40802	6.41239	

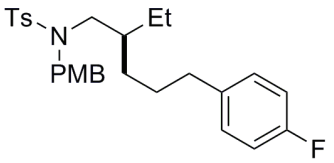


Table 2, entry 1

with (*S*,*S*)-*m*-CF_3-DMPEDA

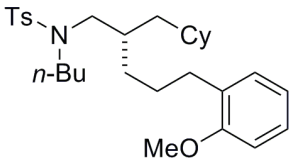
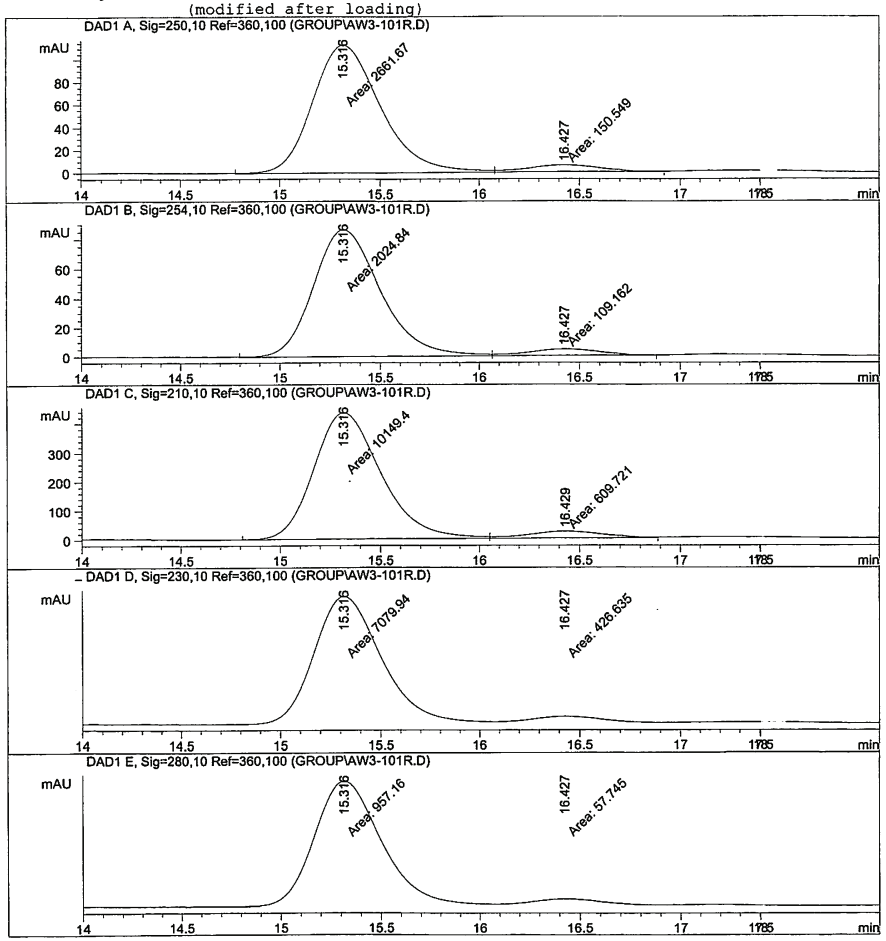


Table 2, entry 2

with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW3-101R.D

```
==========================================================================
Injection Date  : 2/20/2011 12:11:37 PM          Seq. Line :   3
Sample Name     :                                 Location : Vial 10
Acq. Operator   : YL                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 1/7/2011 7:04:34 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 9:41:19 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-101R.D

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.316 MF    0.3922 2661.66992  113.12072  94.6466
   2  16.427 FM    0.4173  150.54866    6.01292   5.3534

Totals :                  2812.21858  119.13364

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.316 MF    0.3905 2024.84436   86.42067  94.8846
   2  16.427 FM    0.4074  109.16243    4.46603   5.1154

Totals :                  2134.00679   90.88670

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.316 MF    0.3898 1.01494e4   433.99777  94.3330
   2  16.429 FM    0.4291  609.72095   23.68450   5.6670

Totals :                  1.07591e4   457.68227

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.316 MF    0.3916 7079.93896  301.32739  94.3165
   2  16.427 FM    0.4286  426.63519   16.58960   5.6835

Totals :                  7506.57416  317.91699

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.316 MF    0.3897  957.16010   40.93772  94.3103
   2  16.427 FM    0.4287   57.74496    2.24479   5.6897

Totals :                  1014.90506   43.18251
```

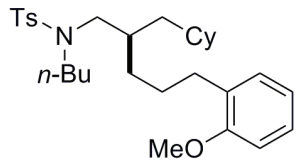
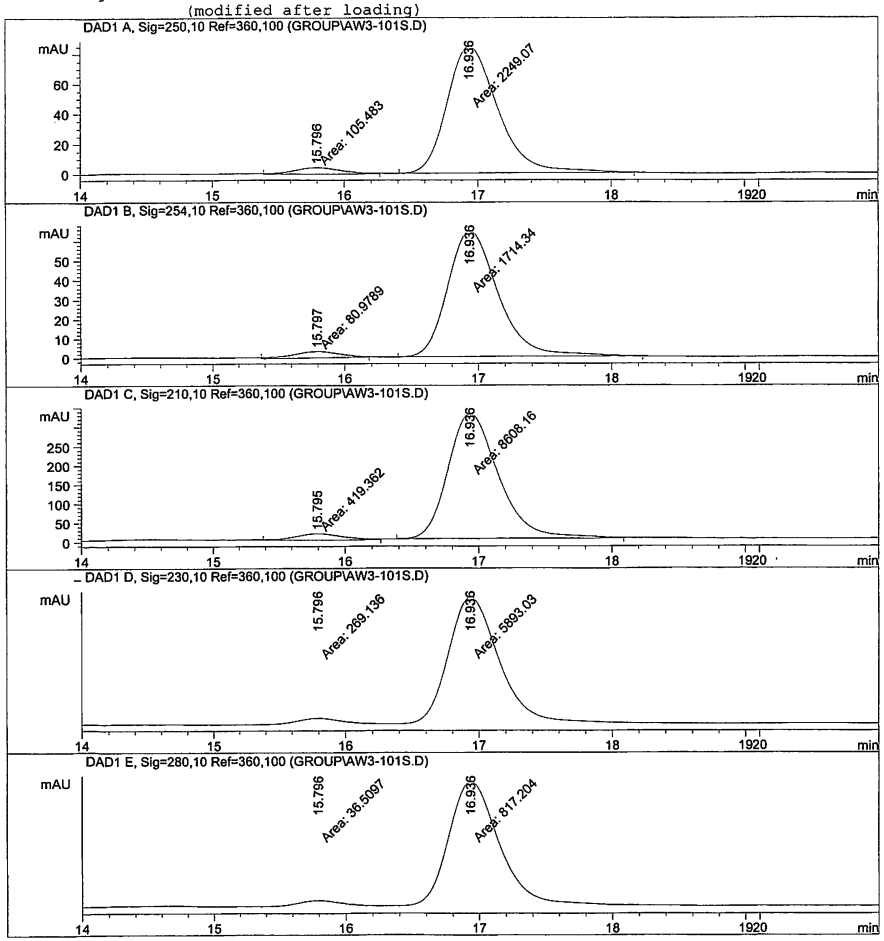


Table 2, entry 2

with (*S*,*S*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW3-101S.D

```
==================================================================
Injection Date  : 2/20/2011 11:40:22 AM          Seq. Line :   2
Sample Name     :                                 Location : Vial 9
Acq. Operator   : YL                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-01-30.M
Last changed    : 1/7/2011 7:04:34 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\YL-AD10.M
Last changed    : 3/15/2012 3:40:26 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-101S.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.796	MM	0.4095	105.48280	4.29328	4.4800
2	16.936	MM	0.4421	2249.06592	84.79340	95.5200
Totals :				2354.54871	89.08668	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.797	MM	0.4079	80.97886	3.30845	4.5106
2	16.936	MM	0.4409	1714.33911	64.80683	95.4894
Totals :				1795.31797	68.11528	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.795	MM	0.4177	419.36249	16.73386	4.6454
2	16.936	MM	0.4399	8608.16309	326.13666	95.3546
Totals :				9027.52557	342.87052	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.796	MM	0.4010	269.13647	11.18549	4.3676
2	16.936	MM	0.4353	5893.02686	225.61151	95.6324
Totals :				6162.16333	236.79700	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.796	MM	0.4020	36.50970	1.51383	4.2766
2	16.936	MM	0.4419	817.20361	30.82416	95.7234
Totals :				853.71331	32.33799	

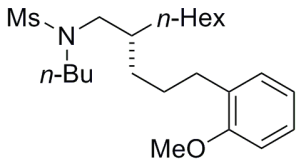
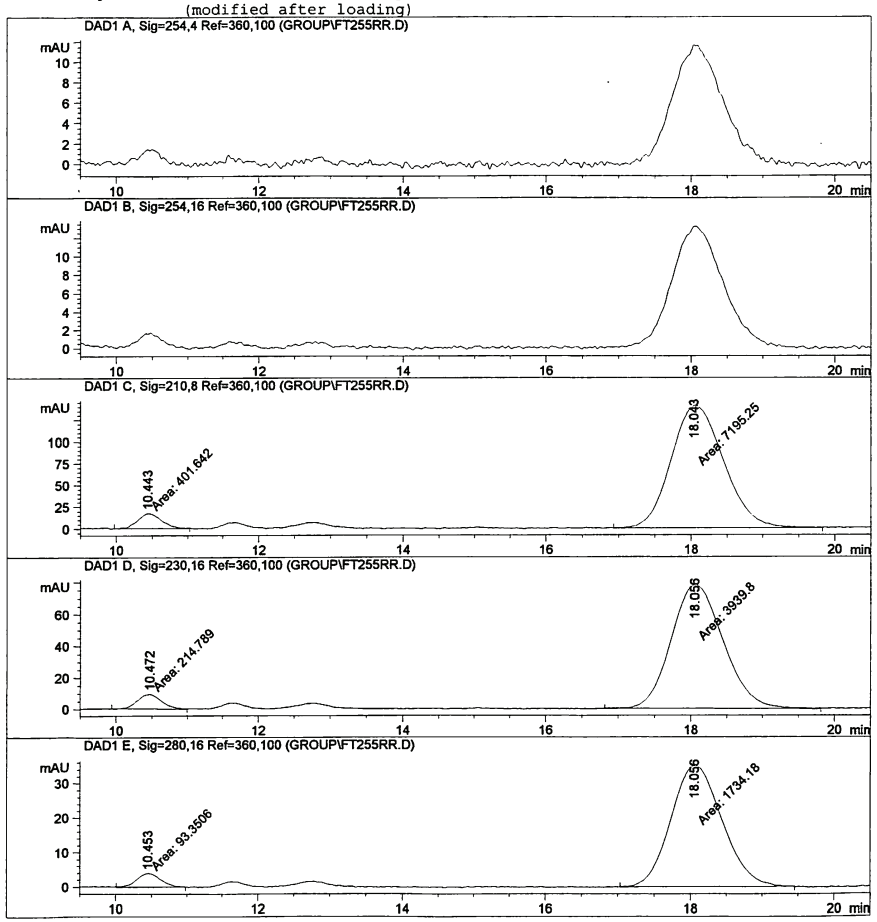


Table 2, entry 3

with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\FT255RR.D

```
==========================================================================
Injection Date  : 9/8/2010 6:15:37 PM          Seq. Line :  10
Sample Name     :                               Location : Vial 72
Acq. Operator   : jtm                             Inj :  1
Acq. Instrument : Instrument 1                Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ASH-0530.M
Last changed    : 6/16/2009 7:52:28 AM by SZ
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0130.M
Last changed    : 3/16/2012 8:01:14 AM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT255RR.D

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.443	MM	0.3891	401.64151	17.20582	5.2869
2	18.043	MM	0.8488	7195.25049	141.28877	94.7131
Totals :				7596.89200	158.49459	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.472	MM	0.3905	214.78893	9.16675	5.1699
2	18.056	MM	0.8436	3939.79663	77.83890	94.8301
Totals :				4154.58556	87.00565	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

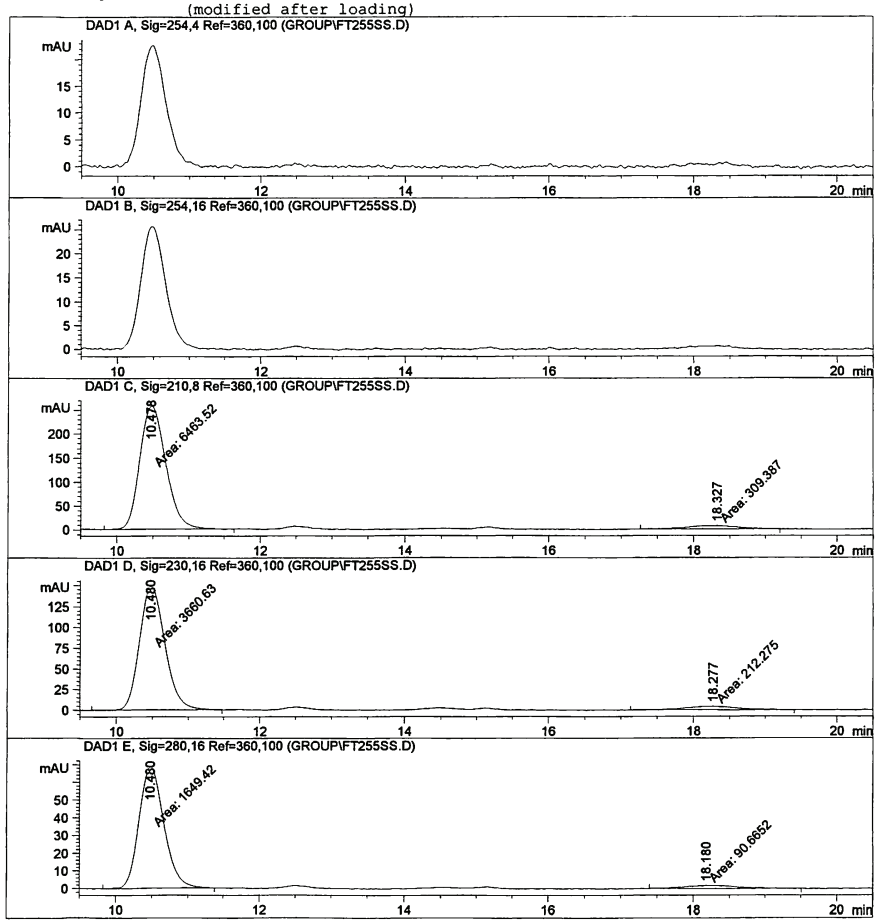
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.453	MM	0.3862	93.35056	4.02850	5.1080
2	18.056	MM	0.8257	1734.18396	35.00381	94.8920
Totals :				1827.53452	39.03231	

Results obtained with enhanced integrator!

```
==========================================================================
                          *** End of Report ***
```

Data File C:\HPCHEM\1\DATA\GROUP\FT255SS.D

```
==================================================================
Injection Date  : 9/8/2010 5:44:25 PM            Seq. Line :   9
Sample Name     :                                 Location : Vial 71
Acq. Operator   : jtm                                Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ASH-0530.M
Last changed    : 6/16/2009 7:52:28 AM by SZ
Analysis Method : C:\HPCHEM\1\METHODS\ADH-3060.M
Last changed    : 3/15/2012 2:53:30 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT255SS.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.478 MM    0.4202 6463.51709  256.35535  95.4320
   2  18.327 MM    0.7542  309.38730    6.83696   4.5680

Totals :                  6772.90439  263.19231

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.480 MM    0.4100 3660.62646  148.78825  94.5190
   2  18.277 MM    0.8146  212.27452    4.34292   5.4810

Totals :                  3872.90099  153.13118

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.480 MM    0.4007 1649.42175   68.60393  94.7896
   2  18.180 MM    0.8105   90.66521    1.86442   5.2104

Totals :                  1740.08697   70.46835

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

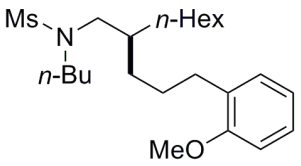


Table 2, entry 3

with (*S*,*S*)-*m*-CF_3-DMPEDA

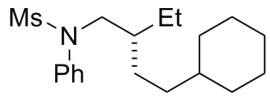
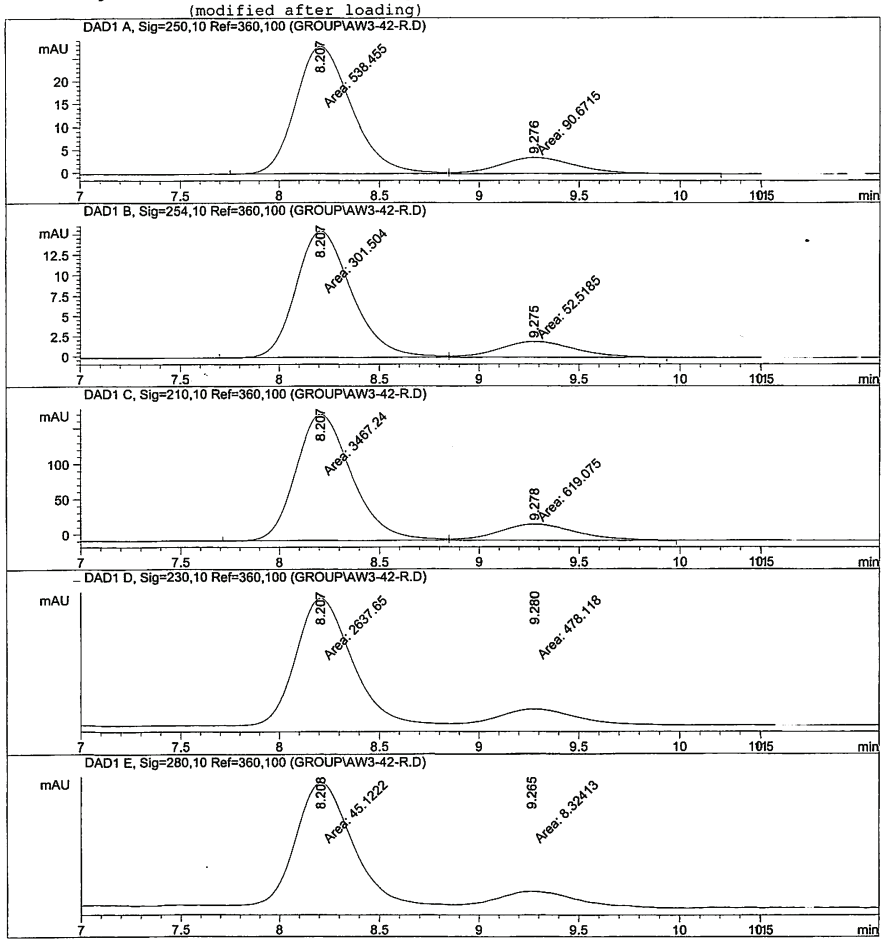


Table 2, entry 4

with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW3-42-R.D

```
==================================================================
Injection Date  : 1/23/2011 11:06:59 AM          Seq. Line :   3
Sample Name     :                                 Location : Vial 7
Acq. Operator   : JTM                                 Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OJ-05-30.M
Last changed    : 4/17/2009 6:56:58 PM by GROUP
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 9:47:55 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-42-R.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.207 MF    0.3225  538.45520   27.82458  85.5877
   2   9.276 FM    0.4346   90.67149    3.47747  14.4123

Totals :                   629.12669   31.30205

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.207 MF    0.3233  301.50403   15.54405  85.1652
   2   9.275 FM    0.4446   52.51852    1.96864  14.8348

Totals :                   354.02254   17.51270

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.207 MF    0.3236 3467.23730  178.59734  84.8500
   2   9.278 FM    0.4498  619.07483   22.93786  15.1500

Totals :                  4086.31213  201.53520

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.207 MF    0.3267 2637.64624  134.56590  84.6549
   2   9.280 FM    0.4599  478.11755   17.32601  15.3451

Totals :                  3115.76379  151.89191

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.208 MF    0.3324   45.12221    2.26224  84.4253
   2   9.265 FM    0.4722    8.32413 2.93776e-1  15.5747

Totals :                   53.44634    2.55602
```

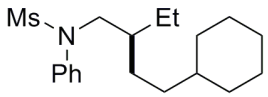
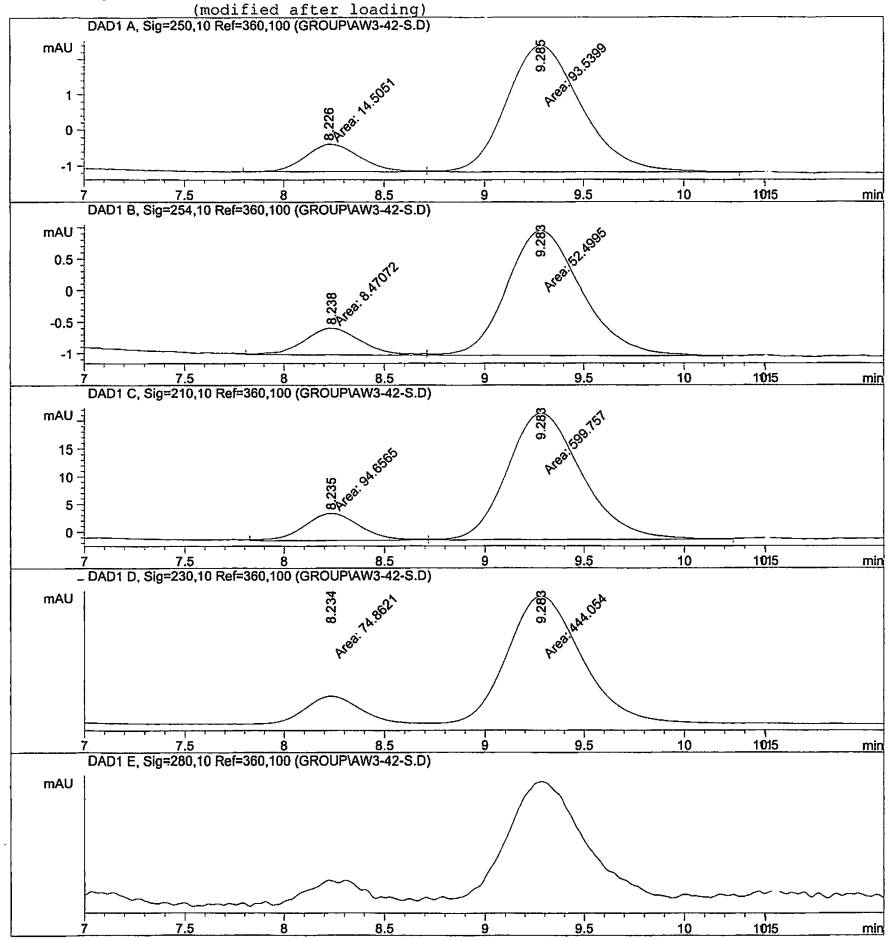


Table 2, entry 4

with (*S*,*S*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW3-42-S.D

```
==================================================================
Injection Date  : 1/23/2011 10:35:47 AM          Seq. Line :   2
Sample Name     :                                 Location : Vial 6
Acq. Operator   : JTM                                 Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OJ-05-30.M
Last changed    : 4/17/2009 6:56:58 PM by GROUP
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 9:51:30 AM by CE
                  (modified after loading)
```

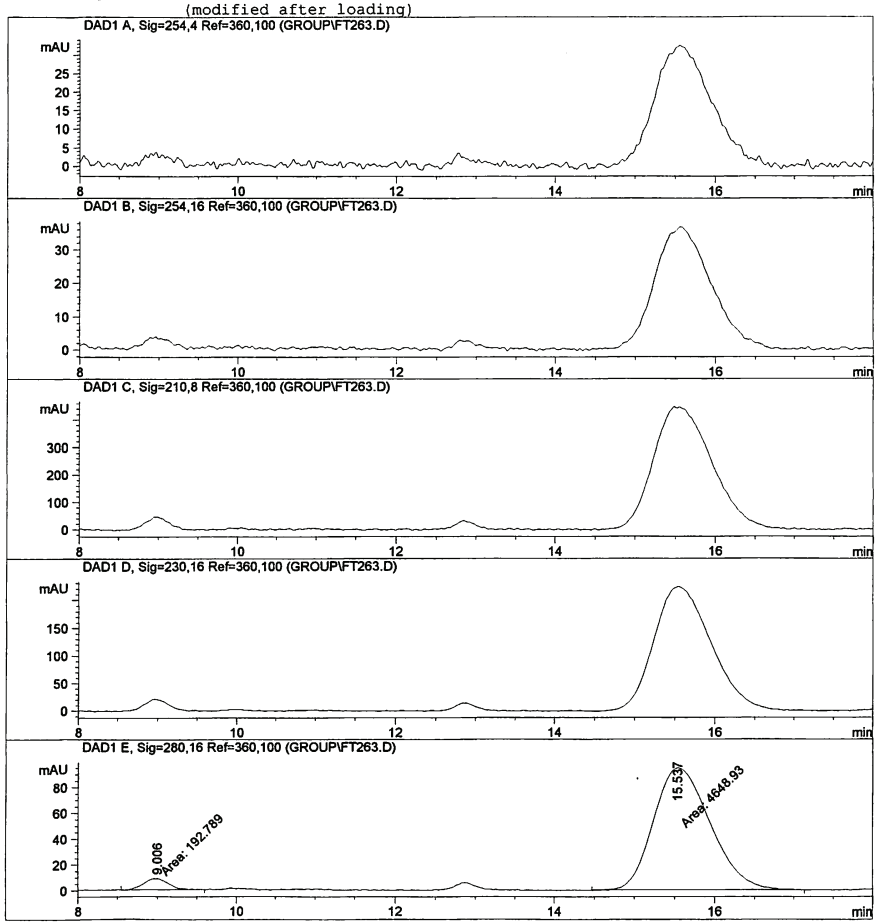


Data File C:\HPCHEM\1\DATA\GROUP\AW3-42-S.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.226 MF    0.3170   14.50510 7.62665e-1  13.4251
   2   9.285 FM    0.4390   93.53989    3.55094  86.5749

Totals :                   108.04499    4.31361

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.238 MF    0.3300    8.47072 4.27860e-1  13.8932
   2   9.283 FM    0.4391   52.49954    1.99285  86.1068

Totals :                    60.97026    2.42071

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.235 MF    0.3246   94.65654    4.85949  13.6311
   2   9.283 FM    0.4388  599.75708   22.78204  86.3689

Totals :                   694.41362   27.64153

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.234 MF    0.3304   74.86207    3.77580  14.4266
   2   9.283 FM    0.4372  444.05356   16.92853  85.5734

Totals :                   518.91563   20.70433

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

==================================================================
                        *** End of Report ***
```

Data File C:\HPCHEM\1\DATA\GROUP\FT263.D

```
Injection Date  : 9/27/2010 5:46:01 PM          Seq. Line :   5
Sample Name     :                               Location : Vial 84
Acq. Operator   : JTM                              Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ASH-0530.M
Last changed    : 6/16/2009 7:52:28 AM by SZ
Analysis Method : C:\HPCHEM\1\METHODS\ADH-3060.M
Last changed    : 3/15/2012 2:56:50 PM by JTM
                  (modified after loading)
```

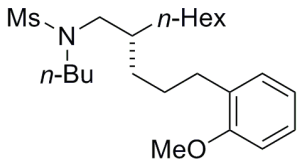


Data File C:\HPCHEM\1\DATA\GROUP\FT263.D

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.006 MM    0.3667  192.78883    8.76340   3.9818
   2  15.537 MM    0.8175 4648.93359   94.77598  96.0182

Totals :                  4841.72243  103.53938

 Results obtained with enhanced integrator!
```

*** End of Report ***



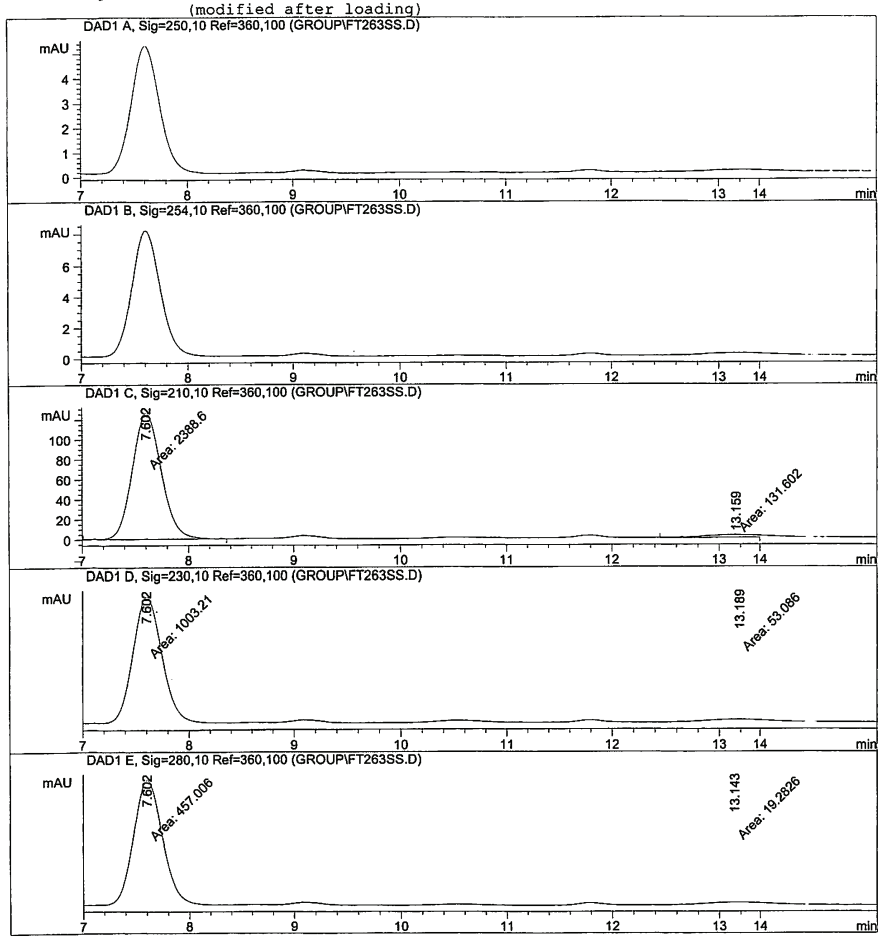
eq 7

with (*R*,*R*)-*m*-CF_3-DMPEDA

S-80

Data File C:\HPCHEM\1\DATA\GROUP\FT263SS.D

```
Injection Date  : 10/4/2010 5:03:33 PM          Seq. Line :  14
Sample Name     :                               Location : Vial 60
Acq. Operator   : JTM                             Inj :   1
Acq. Instrument : Instrument 1                Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AS-05-30.M
Last changed    : 10/4/2010 4:40:59 PM by JTM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\YL-AD10.M
Last changed    : 3/15/2012 3:50:52 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\FT263SS.D

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.602	MM	0.3161	2388.59888	125.93319	94.7781
2	13.159	MM	0.7768	131.60185	2.82341	5.2219
Totals :				2520.20073	128.75660	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.602	MM	0.3161	1003.21118	52.88886	94.9743
2	13.189	MM	0.7341	53.08597	1.20522	5.0257
Totals :				1056.29715	54.09408	

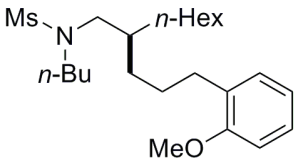
Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.602	MM	0.3156	457.00638	24.13168	95.9515
2	13.143	MM	0.6447	19.28262	4.98468e-1	4.0485
Totals :				476.28900	24.63014	

Results obtained with enhanced integrator!

*** End of Report ***

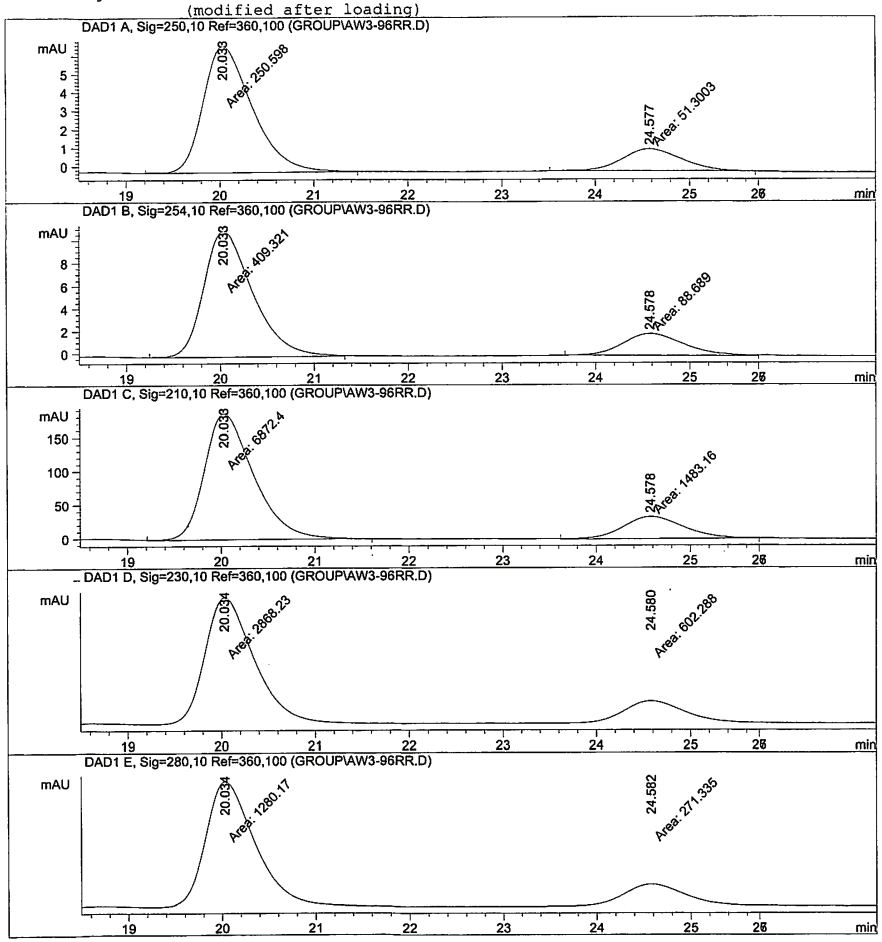


eq 7

with (*S*,*S*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW3-96RR.D

Injection Date	: 2/16/2011 9:09:40 AM	Seq. Line	: 4
Sample Name	:	Location	: Vial 10
Acq. Operator	: YL	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 15 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\OJ-05-30.M		
Last changed	: 4/17/2009 6:56:58 PM by GROUP		
Analysis Method	: C:\HPCHEM\1\METHODS\YL-AD10.M		
Last changed	: 3/15/2012 3:54:52 PM by CE		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\AW3-96RR.D

Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.033	MM	0.6078	250.59778	6.87139	83.0074
2	24.577	MM	0.7166	51.30028	1.19319	16.9926
Totals :				301.89806	8.06458	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.033	MM	0.6103	409.32141	11.17798	82.1913
2	24.578	MM	0.7511	88.68899	1.96796	17.8087
Totals :				498.01040	13.14594	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.033	MM	0.6131	6872.40234	186.83118	82.2494
2	24.578	MM	0.7526	1483.16321	32.84500	17.7506
Totals :				8355.56555	219.67617	

Results obtained with enhanced integrator!

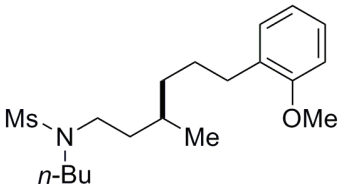
Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.034	MM	0.6112	2868.22559	78.21687	82.6456
2	24.580	MM	0.7358	602.28784	13.64270	17.3544
Totals :				3470.51343	91.85957	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.034	MM	0.6079	1280.17114	35.09542	82.5115
2	24.582	MM	0.7438	271.33539	6.08005	17.4885
Totals :				1551.50653	41.17547	

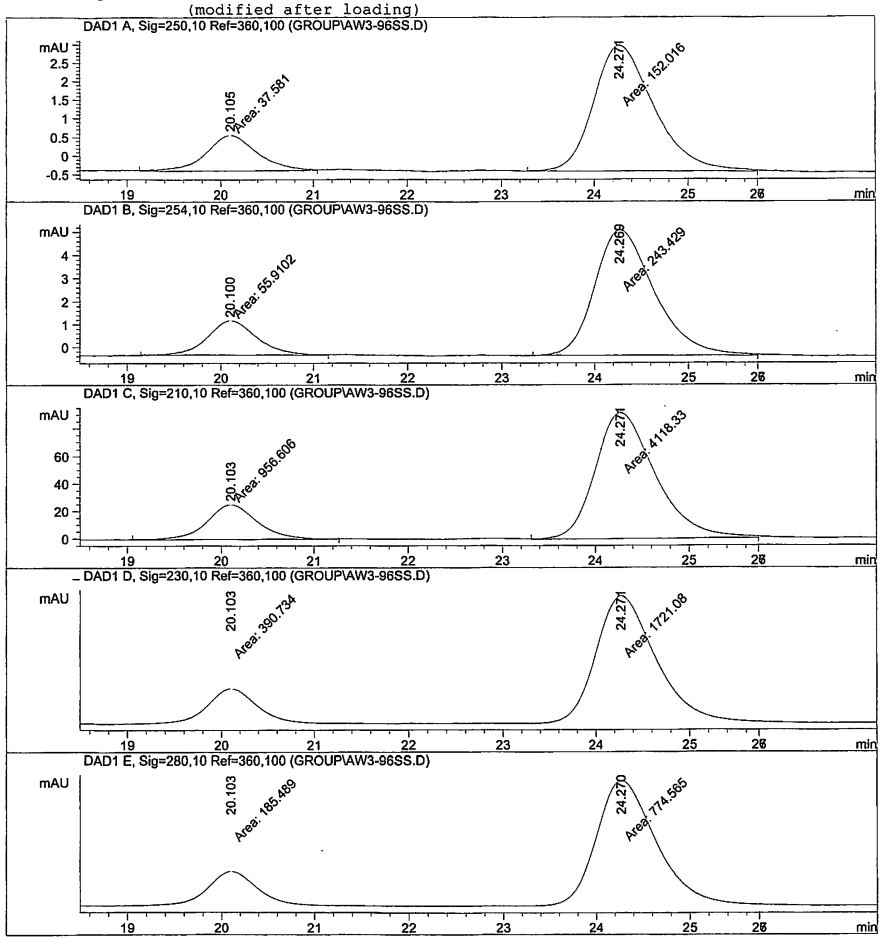


eq 8

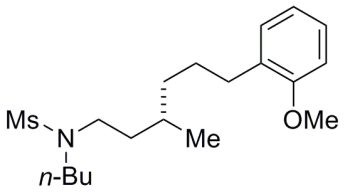
with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW3-96SS.D

```
==========================================================================
Injection Date  : 2/16/2011 8:38:27 AM          Seq. Line :   3
Sample Name     :                               Location : Vial 9
Acq. Operator   : YL                               Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OJ-05-30.M
Last changed    : 4/17/2009 6:56:58 PM by GROUP
Analysis Method : C:\HPCHEM\1\METHODS\YL-AD10.M
Last changed    : 3/15/2012 3:58:09 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW3-96SS.D



eq 8

with (*S*,*S*)-*m*-CF_3-DMPEDA

```
==========================================================
                    Area Percent Report
==========================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.105	MM	0.6507	37.58096	9.62526e-1	19.8215
2	24.271	MM	0.7476	152.01611	3.38901	80.1785
Totals :				189.59707	4.35153	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.100	MM	0.6187	55.91017	1.50611	18.6778
2	24.269	MM	0.7420	243.42947	5.46814	81.3222
Totals :				299.33965	6.97425	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.103	MM	0.6304	956.60577	25.28915	18.8496
2	24.271	MM	0.7495	4118.33496	91.57951	81.1504
Totals :				5074.94073	116.86866	

Results obtained with enhanced integrator!

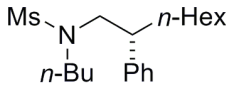
Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.103	MM	0.6156	390.73367	10.57792	18.5023
2	24.271	MM	0.7465	1721.07568	38.42632	81.4977
Totals :				2111.80936	49.00423	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.103	MM	0.6421	185.48911	4.81465	19.3207
2	24.270	MM	0.7492	774.56476	17.23188	80.6793
Totals :				960.05386	22.04654	

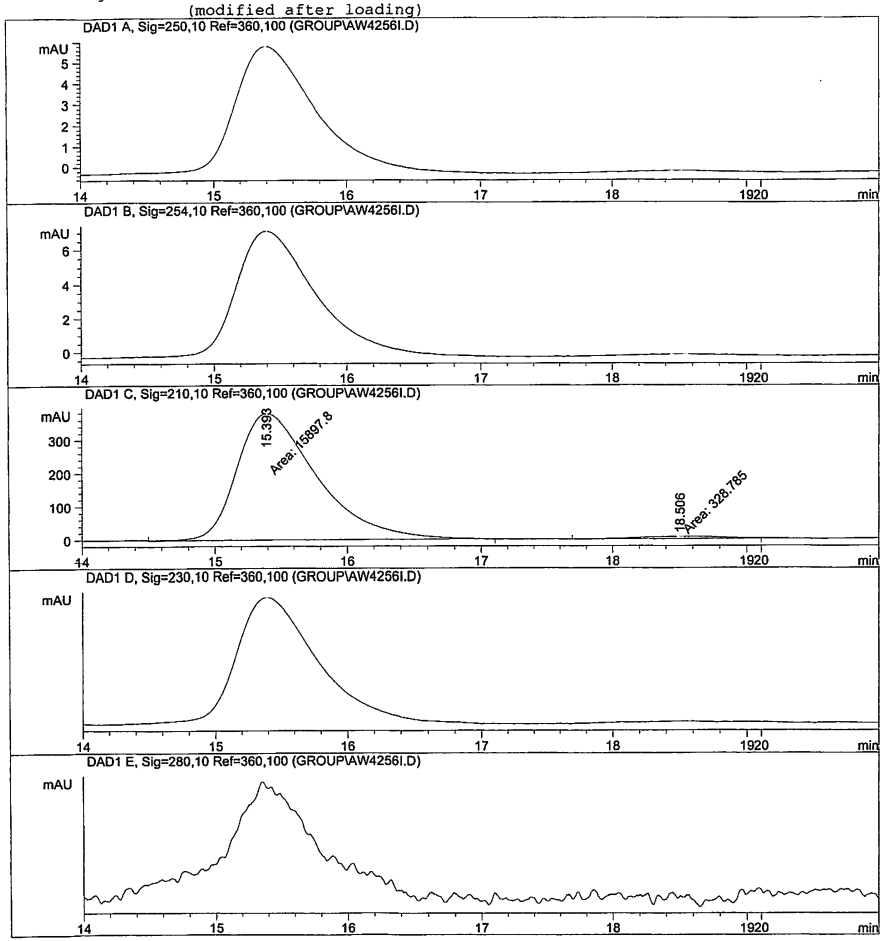


eq 9

with (*R*,*R*)-*m*-CF_3-DMPEDA

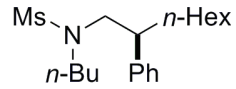
Data File C:\HPCHEM\1\DATA\GROUP\AW4256I.D

```
==================================================================
Injection Date  : 12/18/2011 2:48:50 PM          Seq. Line :  11
Sample Name     :                                 Location : Vial 14
Acq. Operator   : JTM                                Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AS-02-30.M
Last changed    : 12/18/2011 2:48:04 PM by JTM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 9:58:18 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4256I.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100


Signal 2: DAD1 B, Sig=254,10 Ref=360,100


Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.393 MM    0.7003 1.58978e4   378.33545  97.9738
   2  18.506 MM    0.8001  328.78455    6.84874   2.0262

Totals :                  1.62266e4   385.18419

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100


Signal 5: DAD1 E, Sig=280,10 Ref=360,100

==================================================================
                        *** End of Report ***
```

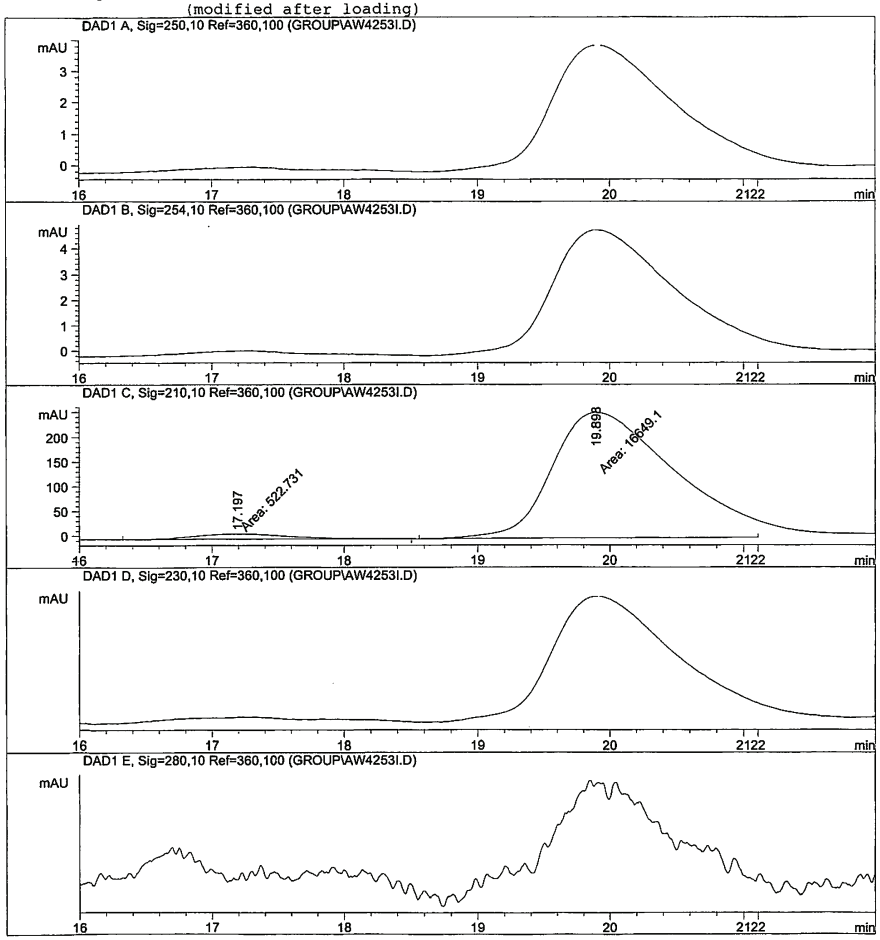


eq 9

with (*S*,*S*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW4253I.D

```
Injection Date  : 12/18/2011 3:15:02 PM          Seq. Line :  12
Sample Name     :                                 Location : Vial 15
Acq. Operator   : JTM                                 Inj :  1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AS-02-30.M
Last changed    : 12/18/2011 2:48:04 PM by JTM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 10:00:31 AM by CE
                  (modified after loading)
```



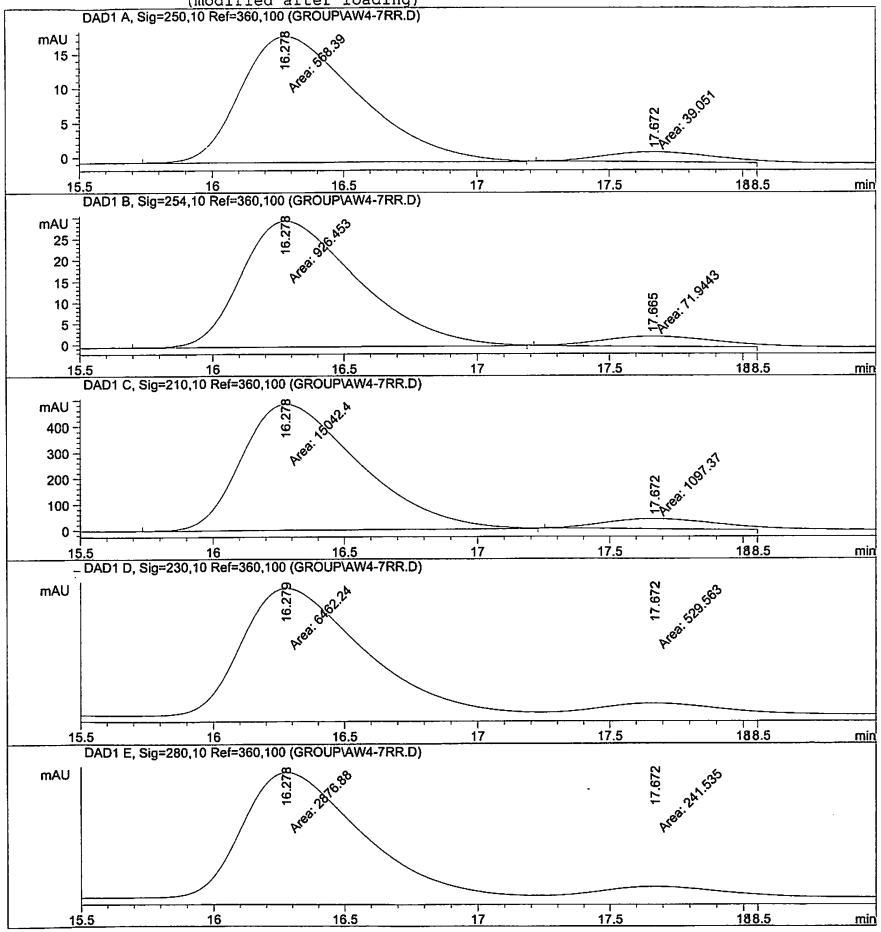
Data File C:\HPCHEM\1\DATA\GROUP\AW4253I.D

```
                              Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100


Signal 2: DAD1 B, Sig=254,10 Ref=360,100


Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.197 MM    0.8357  522.73090   10.42530   3.0441
   2  19.898 MF    1.0944 1.66491e4   253.55196  96.9559

Totals :                  1.71719e4   263.97726

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100


Signal 5: DAD1 E, Sig=280,10 Ref=360,100

                           *** End of Report ***
```

S-85

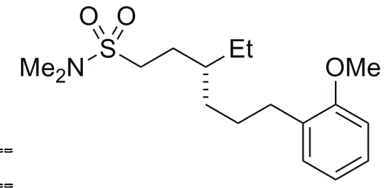
Data File C:\HPCHEM\1\DATA\GROUP\AW4-7RR.D

```
==================================================================
Injection Date  : 6/18/2011 7:45:41 AM          Seq. Line :   3
Sample Name     :                                Location : Vial 32
Acq. Operator   : NB                                Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OD-05-30.M
Last changed    : 5/5/2011 12:12:20 PM by NB
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 10:07:43 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4-7RR.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.278 MM    0.5192  568.39008   18.24712  93.5712
   2  17.672 MM    0.4693   39.05103    1.38698   6.4288

Totals :                   607.44110   19.63410

 Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.278 MM    0.5182  926.45325   29.79666  92.7940
   2  17.665 MM    0.5016   71.94431    2.39066   7.2060

Totals :                   998.39755   32.18732

 Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.278 MM    0.5179 1.50424e4   484.07565  93.2008
   2  17.672 MM    0.4826 1097.37256   37.89697   6.7992

Totals :                  1.61398e4   521.97262

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.279 MM    0.5290 6462.23975  203.60910  92.4259
   2  17.672 MM    0.5252  529.56348   16.80620   7.5741

Totals :                  6991.80322  220.41530

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.278 MM    0.5160 2876.87964   92.92225  92.2546
   2  17.672 MM    0.5264  241.53452    7.64759   7.7454

Totals :                  3118.41415  100.56983
```

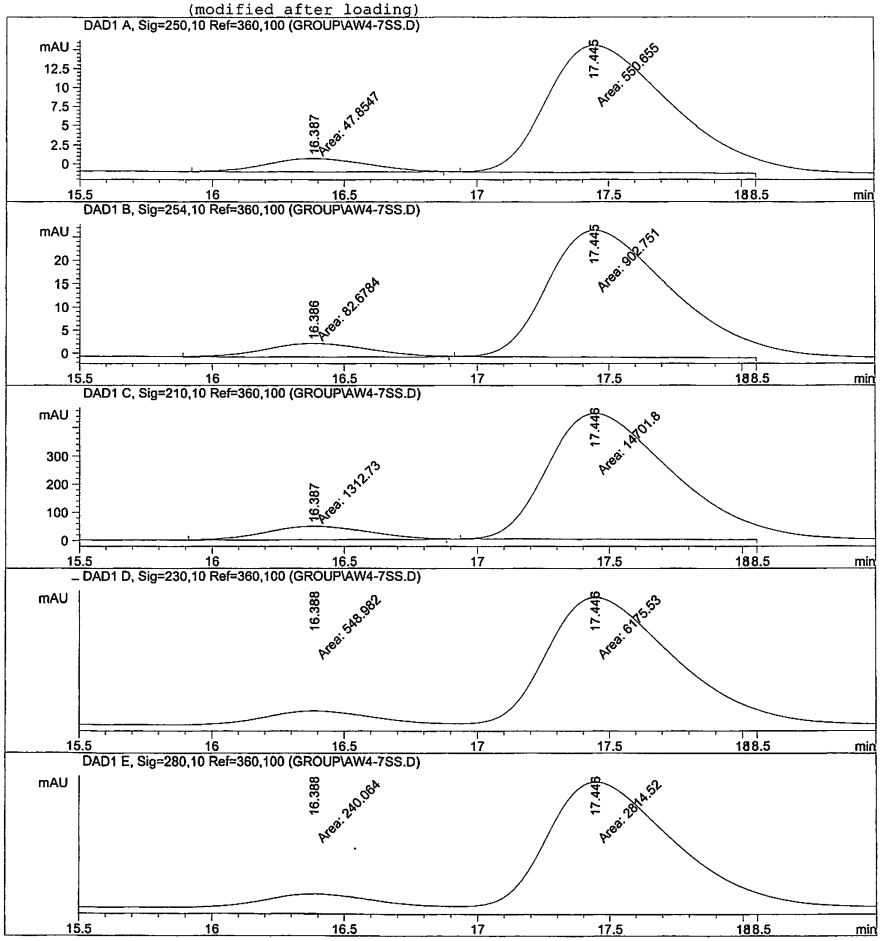


eq 10

with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW4-7SS.D

```
==================================================================
Injection Date  : 6/18/2011 7:14:24 AM          Seq. Line :   2
Sample Name     :                               Location : Vial 31
Acq. Operator   : NB                               Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OD-05-30.M
Last changed    : 5/5/2011 12:12:20 PM by NB
Analysis Method : C:\HPCHEM\1\METHODS\HDOD1.M
Last changed    : 3/15/2012 5:04:39 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4-7SS.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.387	MM	0.4484	47.85474	1.77876	7.9956
2	17.445	MM	0.5501	550.65503	16.68246	92.0044
Totals :				598.50977	18.46122	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.386	MM	0.4653	82.67843	2.96174	8.3901
2	17.445	MM	0.5517	902.75092	27.26981	91.6099
Totals :				985.42934	30.23154	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.387	MM	0.4562	1312.72607	47.95905	8.1971
2	17.446	MM	0.5517	1.47018e4	444.15845	91.8029
Totals :				1.60145e4	492.11750	

Results obtained with enhanced integrator!

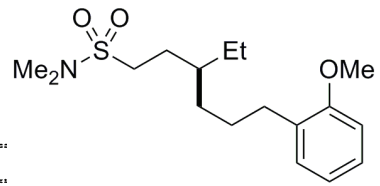
Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.388	MM	0.4555	548.98169	20.08522	8.1639
2	17.446	MM	0.5517	6175.52930	186.54672	91.8361
Totals :				6724.51099	206.63194	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.388	MM	0.4477	240.06416	8.93773	7.8591
2	17.446	MM	0.5497	2814.51880	85.33121	92.1409
Totals :				3054.58296	94.26894	

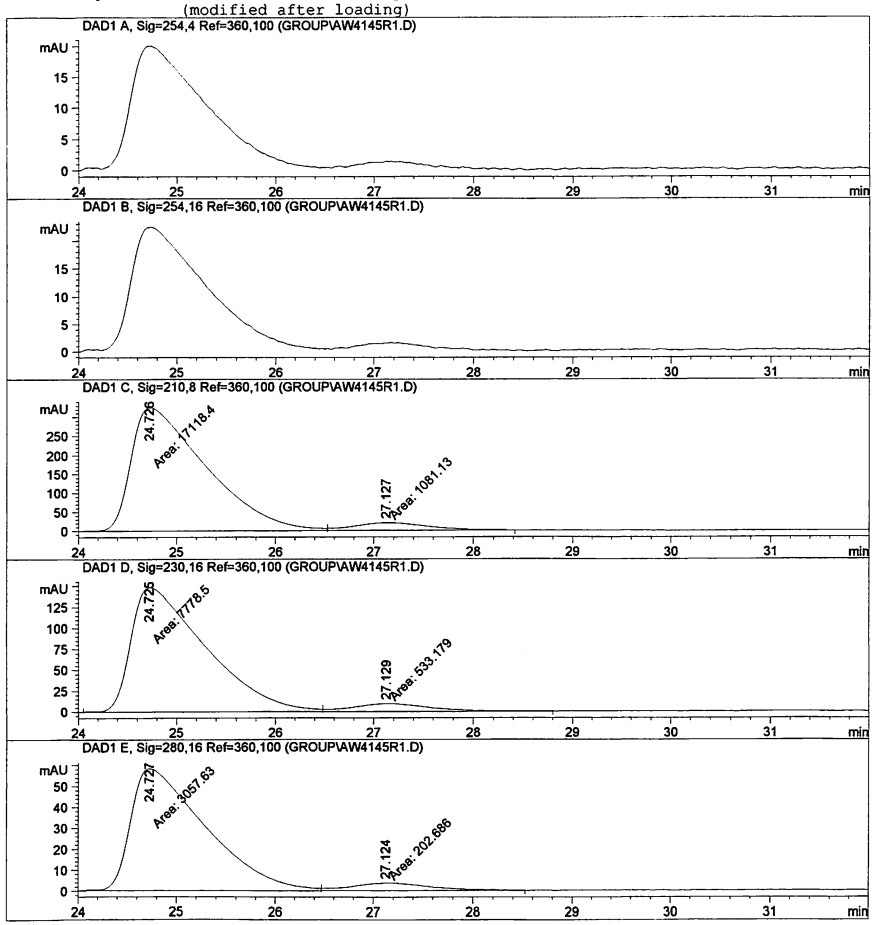


eq 10

with (*S*,*S*)-*m*-CF_3-DMPEDA

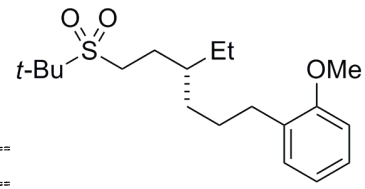
Data File C:\HPCHEM\1\DATA\GROUP\AW4145R1.D

```
==================================================================
Injection Date  : 9/20/2011 11:52:23 AM          Seq. Line :   3
Sample Name     :                                 Location : Vial 83
Acq. Operator   : SN                                  Inj :   1
Acq. Instrument : Instrument 1                Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\IA-0140.M
Last changed    : 5/19/2011 9:18:58 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0120.M
Last changed    : 3/15/2012 5:17:33 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4145R1.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.726 MF    0.8745 1.71184e4   326.25052  94.0596
   2  27.127 FM    0.8734 1081.13184   20.63085   5.9404

Totals :                  1.81995e4   346.88137

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.725 MF    0.8753 7778.49561  148.11688  93.5852
   2  27.129 FM    0.9292  533.17920    9.56356   6.4148

Totals :                  8311.67480  157.68045

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.727 MF    0.8737 3057.63403   58.32730  93.7832
   2  27.124 FM    0.9089  202.68625    3.71649   6.2168

Totals :                  3260.32028   62.04380

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```



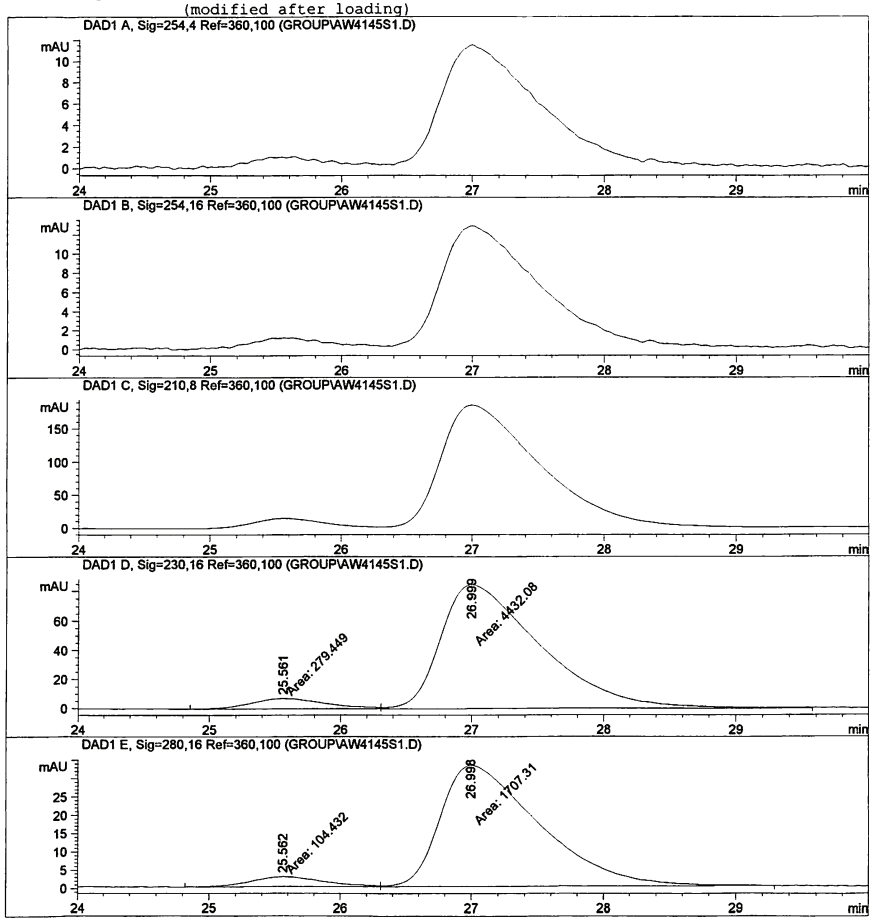
eq 11

with (*R*,*R*)-*m*-CF_3-DMPEDA

S-88

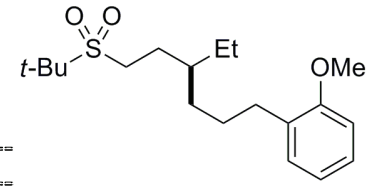
Data File C:\HPCHEM\1\DATA\GROUP\AW4145S1.D

```
==================================================================
Injection Date  : 9/20/2011 11:11:02 AM          Seq. Line :   2
Sample Name     :                                 Location : Vial 82
Acq. Operator   : SN                                   Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\IA-0140.M
Last changed    : 5/19/2011 9:18:58 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0130.M
Last changed    : 3/16/2012 8:05:21 AM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4145S1.D

```
==============================================================
                      Area Percent Report
==============================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100


Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.561 MF    0.6578  279.44861    7.08071   5.9312
   2  26.999 FM    0.8727 4432.07568   84.63859  94.0688

Totals :                  4711.52429   91.71930

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.562 MF    0.6364  104.43195    2.73509   5.7642
   2  26.998 FM    0.8610 1707.31458   33.04961  94.2358

Totals :                  1811.74652   35.78470

 Results obtained with enhanced integrator!
==============================================================
                      *** End of Report ***
```



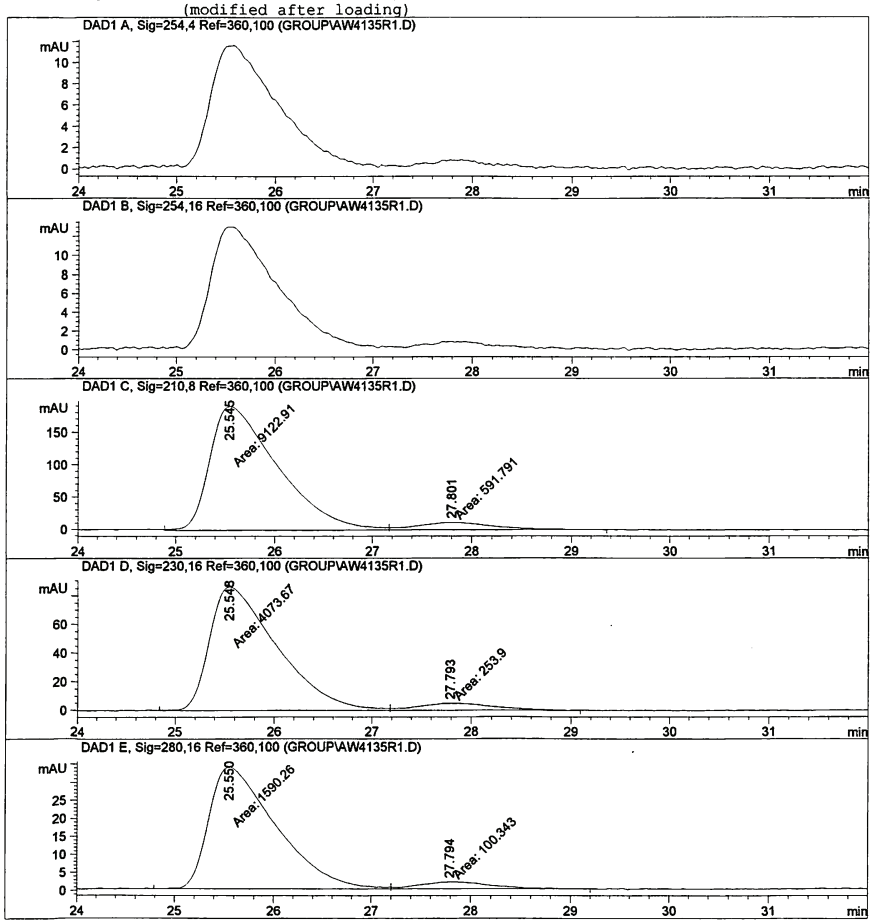
eq 11

with (*S*,*S*)-*m*-CF_3-DMPEDA

S-89

Data File C:\HPCHEM\1\DATA\GROUP\AW4135R1.D

```
==================================================================
Injection Date  : 9/20/2011 1:13:25 PM          Seq. Line :   5
Sample Name     :                                Location : Vial 85
Acq. Operator   : SN                                Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\IA-0140.M
Last changed    : 9/20/2011 1:50:26 PM by SN
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0120.M
Last changed    : 3/15/2012 5:17:33 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4135R1.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.545 MF    0.7958 9122.91211  191.06412  93.9083
   2  27.801 FM    0.8771  591.79144   11.24574   6.0917

Totals :                  9714.70355  202.30986

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.548 MF    0.7878 4073.67090   86.18327  94.1330
   2  27.793 FM    0.8542  253.89975    4.95401   5.8670

Totals :                  4327.57065   91.13728

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.550 MF    0.7864 1590.25549   33.70442  94.0646
   2  27.794 FM    0.8607  100.34315    1.94312   5.9354

Totals :                  1690.59864   35.64754

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

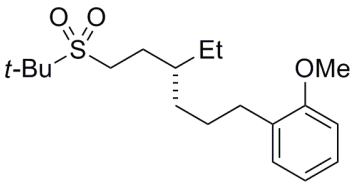


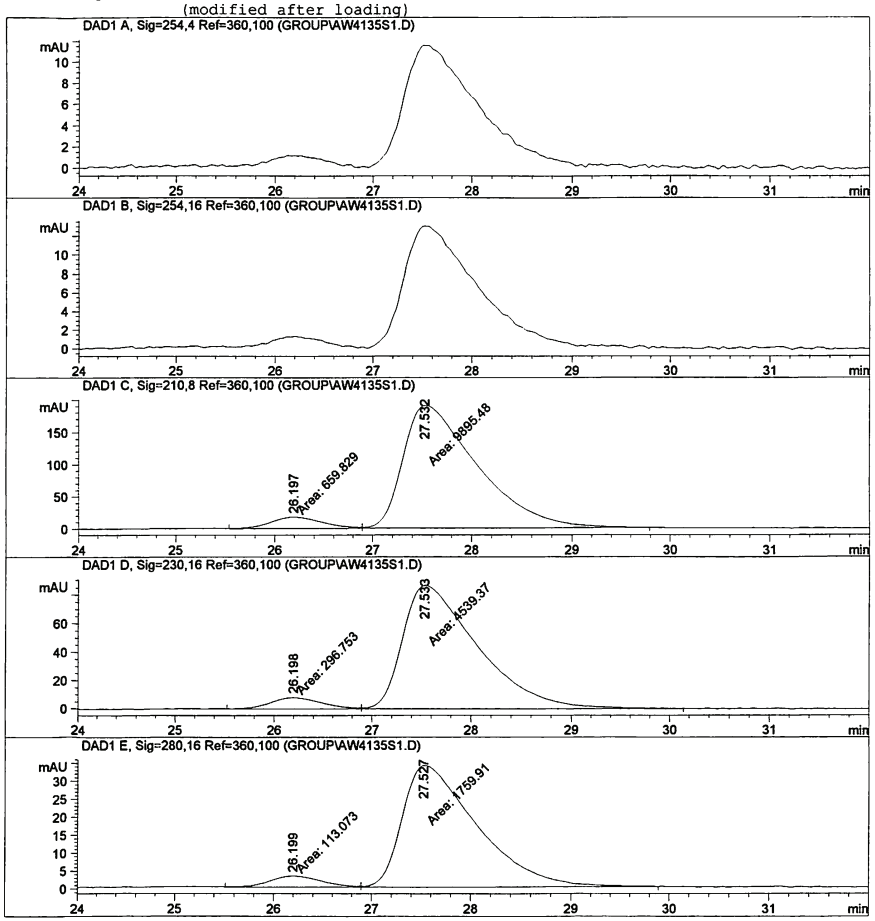
Table 3, entry 1

with (*R*,*R*)-*m*-CF_3-DMPEDA

S-90

Data File C:\HPCHEM\1\DATA\GROUP\AW4135S1.D

```
==================================================================
Injection Date  : 9/20/2011 12:33:38 PM          Seq. Line :   4
Sample Name     :                                 Location : Vial 84
Acq. Operator   : SN                                  Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\IA-0140.M
Last changed    : 9/20/2011 1:11:57 PM by SN
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0120.M
Last changed    : 3/15/2012 5:10:25 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4135S1.D

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  26.197 MF    0.6183  659.82867   17.78742   6.2512
   2  27.532 FM    0.8671 9895.47852  190.19273  93.7488

Totals :                  1.05553e4   207.98016

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  26.198 MF    0.6099  296.75323    8.10926   6.1362
   2  27.533 FM    0.8747 4539.36621   86.49334  93.8638

Totals :                  4836.11945   94.60260

 Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  26.199 MF    0.5987  113.07254    3.14748   6.0370
   2  27.527 FM    0.8676 1759.91003   33.80847  93.9630

Totals :                  1872.98257   36.95595

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

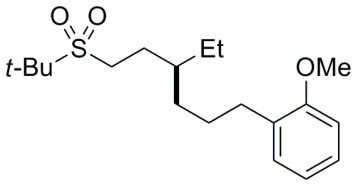


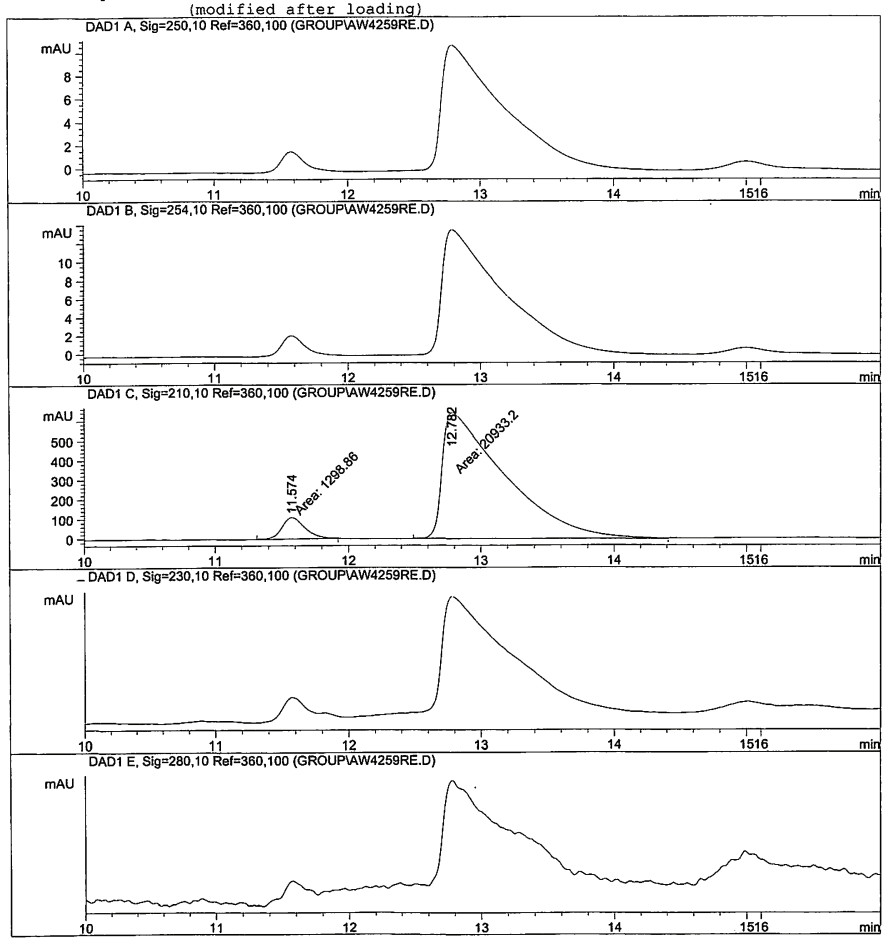
Table 3, entry 1

with (*S*,*S*)-*m*-CF_3-DMPEDA

S-91

Data File C:\HPCHEM\1\DATA\GROUP\AW4259RE.D

```
Injection Date  : 12/19/2011 6:44:25 AM          Seq. Line :   3
Sample Name     :                                Location : Vial 12
Acq. Operator   : JTM                              Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\IB-01-30.M
Last changed    : 12/18/2011 9:53:08 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\HDOD1.M
Last changed    : 3/15/2012 5:27:59 PM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4259RE.D

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100


Signal 2: DAD1 B, Sig=254,10 Ref=360,100


Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.574 MM    0.1978 1298.86377  109.44008   5.8423
   2  12.782 MM    0.5440 2.09332e4   641.35797  94.1577

Totals :                  2.22320e4   750.79805

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100


Signal 5: DAD1 E, Sig=280,10 Ref=360,100
```

*** End of Report ***

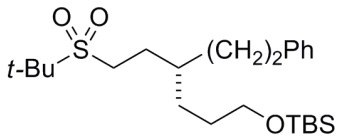
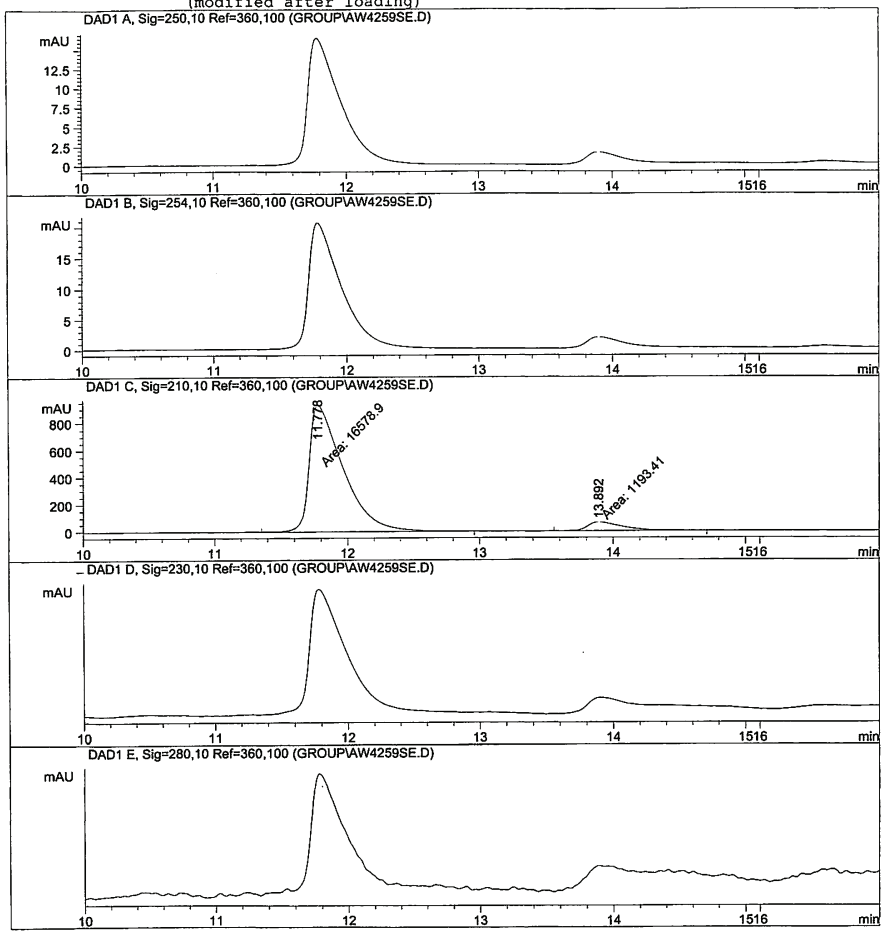


Table 3, entry 2

with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW4259SE.D

```
==========================================================================
Injection Date  : 12/19/2011 6:13:10 AM          Seq. Line :   2
Sample Name     :                                 Location : Vial 11
Acq. Operator   : JTM                                Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\IB-01-30.M
Last changed    : 12/18/2011 9:53:08 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\AD-20-80.M
Last changed    : 3/16/2012 10:13:33 AM by CE
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4259SE.D

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,10 Ref=360,100


Signal 2: DAD1 B, Sig=254,10 Ref=360,100


Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.778 MM    0.2968 1.65789e4   931.02295  93.2850
   2  13.892 MM    0.3053 1193.40845   65.14220   6.7150

Totals :                  1.77723e4   996.16515

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100


Signal 5: DAD1 E, Sig=280,10 Ref=360,100

==========================================================================
                          *** End of Report ***
```

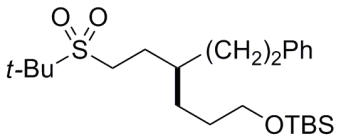
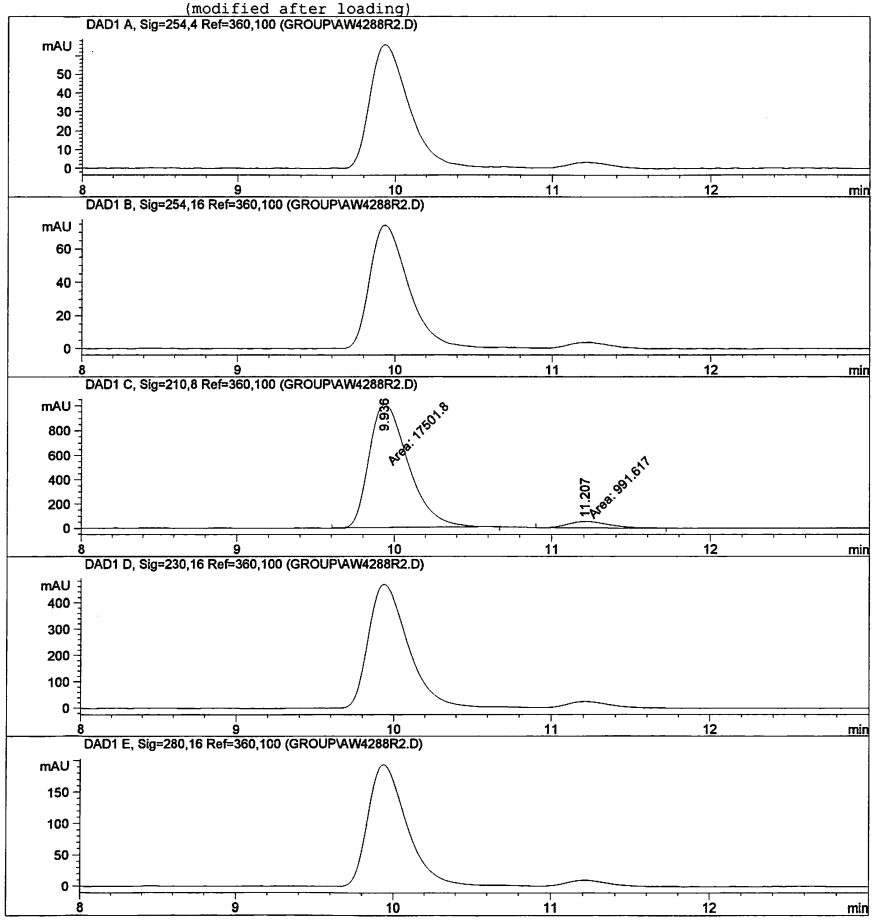


Table 3, entry 2

with (*S*,*S*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW4288R2.D

```
==========================================================================
Injection Date  : 1/22/2012 3:13:49 PM          Seq. Line :   5
Sample Name     :                                Location : Vial 73
Acq. Operator   : JTM                               Inj :  1
Acq. Instrument : Instrument 1                  Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0530.M
Last changed    : 6/18/2011 6:35:43 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0120.M
Last changed    : 3/15/2012 5:17:33 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4288R2.D

```
==========================================================
                      Area Percent Report
==========================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.936 MM    0.2906 1.75018e4  1003.61664  94.6380
   2  11.207 MM    0.3044  991.61719   54.29025   5.3620

Totals :                  1.84934e4  1057.90689

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                        *** End of Report ***
```

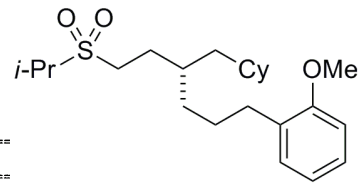
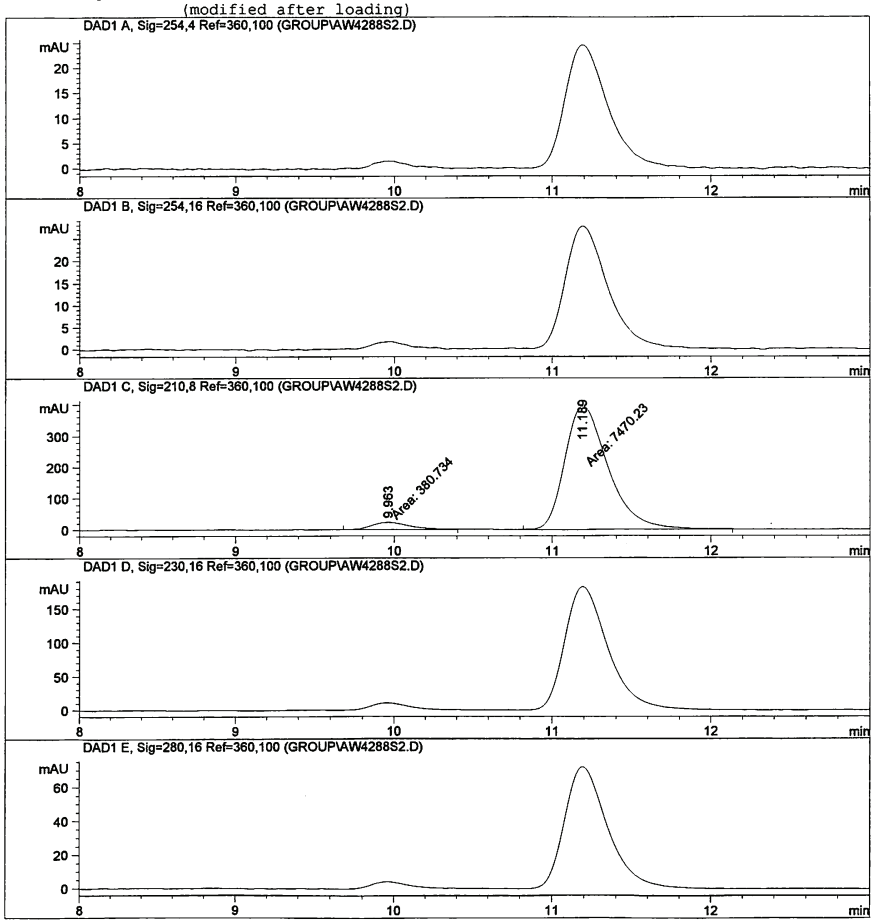


Table 3, entry 3

with (*R*,*R*)-*m*-CF_3-DMPEDA

Data File C:\HPCHEM\1\DATA\GROUP\AW4288S2.D

```
==========================================================================
Injection Date  : 1/22/2012 3:45:03 PM          Seq. Line :   6
Sample Name     :                               Location : Vial 72
Acq. Operator   : JTM                              Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 5 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0530.M
Last changed    : 1/22/2012 4:10:10 PM by JTM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0120.M
Last changed    : 3/15/2012 6:18:31 PM by JTM
                  (modified after loading)
```



Data File C:\HPCHEM\1\DATA\GROUP\AW4288S2.D

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100


Signal 2: DAD1 B, Sig=254,16 Ref=360,100


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.963 MM    0.2790  380.73450   22.74090   4.8495
   2  11.189 MM    0.3153 7470.22607  394.83435  95.1505

Totals :                  7850.96057  417.57525

 Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100


Signal 5: DAD1 E, Sig=280,16 Ref=360,100

==========================================================================
                          *** End of Report ***
```

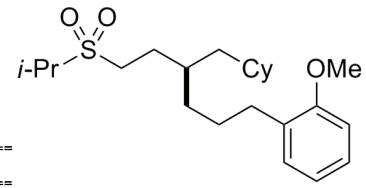


Table 3, entry 3

with (*S*,*S*)-*m*-CF_3-DMPEDA